

# Technical Service Bulletin 909

## Cleaning of Fouled Resins

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Ion exchange resins can become contaminated by various substances during use, leading to a loss of ion exchange capacity. The mechanisms by which contamination occurs and the methods for cleaning the contaminated substances can vary. The purpose of this technical document is to understand the treatment methods for each case to ensure stable operation. If the ion exchange resin is contaminated over a long period, the actual treatment described below may be ineffective, so it is best to reduce contamination of the ion exchange resin through appropriate pretreatment.



### Cautions

1. Familiarize yourself with the Material Safety Data Sheets (SDS) for all chemicals used.
2. Always work in well-ventilated area.
3. Wear proper PPE like tight goggles, nitrile gloves, acid/alkali proof clothing as may be required.
4. Always add acid to water.

### Warnings

1. Do not inhale vapours/fumes.
2. Avoid substance contact.
3. Do not empty acidic/alkaline contents to unspecified drains.

**Please refer the MSDS for First Aid information.**

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### Equipment and Tools Required

Chemical-resistant pumps and hoses for solution circulation

Temperature-controlled heating equipment (for heated cleaning solutions)

pH meter for monitoring solution acidity/alkalinity – portable Hand held

Conductivity meter for assessing rinse effectiveness – portable hand held

Chemical storage containers for cleaning solutions – regeneration tanks can be used too.

Proper waste containment and disposal equipment

Sampling equipment for resin and solution analysis

Calibrated measuring devices for chemical preparation

Emergency eyewash station and safety shower nearby

Resin transfer equipment (e.g., eductor or vacuum system)

Personal protective equipment (PPE) storage and disposal area

Spill containment materials (e.g., absorbent pads, neutralizing agents)

Portable air monitoring equipment for confined space entry

Pressure gauges to monitor system pressure during cleaning

Flow meters to ensure proper circulation rates

Temporary storage tanks for spent cleaning solutions

Labeling materials for identifying chemicals and solutions

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### 1. Contamination of Cation Exchange Resin Column

#### 1.1 Contamination by Iron

##### 1.1.1 Iron Contamination Mechanism

Iron exists as divalent and trivalent inorganic salts or as organic complexes. Divalent iron can be exchanged in the ion exchange resin, but trivalent iron cannot be exchanged as it is insoluble. Trivalent iron deposits in the cation exchange resin, causing performance degradation. To remove deposited iron, acid cleaning or a strong reducing agent should be used. Iron bound to organic matter passes through the cation exchange column and becomes a contaminant in the anion exchange resin. This should be removed along with the organic matter. Manganese has a similar contamination mechanism to iron.

##### 1.1.2 Regeneration Method for Iron-Contaminated Ion Exchange Resin

###### 1) Air Injection and Backwash

When iron with inorganic and organic substances combine to form insoluble precipitates, the following methods are used to remove them:

- (1) Blow compressed air from the bottom of the ion exchange resin column to sufficiently shake the resin layer and loosen any blocked resin layers.
- (2) Perform backwash for 30 minutes to discharge insoluble precipitates.
- (3) Conduct the regeneration process.

###### 2) $\text{Na}_2\text{S}_2\text{O}_4$ Treatment

$\text{Na}_2\text{S}_2\text{O}_4$  is a reducing agent that reduces the trivalent iron deposited in the ion exchange resin to soluble divalent iron. The reduced and soluble iron can be removed during the backwash or regeneration process.

- (1) The pore volume (space between resin particles) is roughly estimated to be half the amount of resin filled. Adjust the water level in the resin column by draining (to be slightly more than the pore volume + the resin layer height).
- (2) Prepare  $\text{Na}_2\text{S}_2\text{O}_4$  so that a 4% solution is formed when mixed with the water above the resin layer. (Do not add water to  $\text{Na}_2\text{S}_2\text{O}_4$ ; add  $\text{Na}_2\text{S}_2\text{O}_4$  to water) During this process, strong odors are emitted, so ventilation is required.  $\text{Na}_2\text{S}_2\text{O}_4$  is dangerous when it comes into contact with eyes and skin, so caution is needed.
- (3) Introduce the concentrated  $\text{Na}_2\text{S}_2\text{O}_4$  solution into the resin column.
- (4) Drain water from the bottom of the resin column equal to the pore volume which is roughly 50% of resin volume.
- (5) Mix the aqueous solution inside the resin column, with a paddle, to spread the  $\text{Na}_2\text{S}_2\text{O}_4$  throughout the resin layer. Note that if air is used for stirring,  $\text{Na}_2\text{S}_2\text{O}_4$  may oxidize and lose its reducing power.
- (6) Mix the aqueous solution inside the resin column, with a paddle, to spread the  $\text{Na}_2\text{S}_2\text{O}_4$  throughout the resin layer. Note that if air is used for stirring,  $\text{Na}_2\text{S}_2\text{O}_4$  may oxidize and lose its reducing power.
- (7) Leave the  $\text{Na}_2\text{S}_2\text{O}_4$  in contact with the resin layer for 12 hours. If this is not feasible, a minimum contact time of 4 hours is recommended.
- (8) Drain the reduced iron and other impurities, rinse with feed water, and perform backwash for 30 minutes.
- (9) Perform regeneration prior to initiating service operation.

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### 3) Hydrochloric Acid Treatment

When the ion exchange resin is severely contaminated, hydrochloric acid (HCl) washing is the only method to restore the exchange capacity of the ion exchange resin. Acid washing is risky due to its potential to damage the piping line, so it should be performed with caution. The resin column and its piping equipment may be damaged by contact with hydrochloric acid. Therefore, hydrochloric acid washing should only be performed after inspecting the equipment and ensuring that it is durable enough to withstand the acid; otherwise, use corrosion-resistant hydrochloric acid. Corrosion-resistant hydrochloric acid is hydrochloric acid mixed with a corrosion inhibitor. The corrosion inhibitor is typically a nitrogen compound that forms a metallic film on the resin column or piping to prevent corrosion. Cleaning procedure is the same as when using  $\text{Na}_2\text{S}_2\text{O}_4$ .

### 1.2 Contamination by Sulfates ( $\text{CaSO}_4$ or $\text{MgSO}_4$ )

#### 1.2.1 Sulfate Contamination Mechanism

When sulfuric acid is used as a regenerant in a cation exchange resin column, insoluble salts (sulfates,  $\text{CaSO}_4$  or  $\text{MgSO}_4$ ) may form due to a reaction between the hardness components of the raw water and the regenerant. When sulfates accumulate in the resin layer, they will slowly dissolve during service, degrading water quality and clogging the micropores of the ion exchange resin, thus inhibiting ion diffusion and reducing exchange capacity.

#### 1.2.2 Regeneration Method for Sulfate-Contaminated Ion Exchange Resin

- 1) Perform a normal cation exchange resin regeneration.
- 2) Charge the column with a 10% sodium citrate solution at 1BV (Bed Volume) in an up-flow direction for 20-30 minutes.
- 3) Allow enough time for the citrate to react with the ion exchange resin by blowing air and soaking for about 12 hours.
- 4) Rinse with 5BV of pure water in a downflow direction.
- 5) Perform backwash and a double regeneration before returning to service operation.

### 1.3 Contamination by Polyelectrolyte

#### 1.3.1 Polyelectrolyte Contamination Mechanism

Contamination by polyelectrolytes can occur when raw water quality deteriorates in winter or when there are problems with pretreatment. When contamination becomes severe, the resin layer becomes blocked and sticky, leading to poor flow.

#### 1.3.2 Regeneration Method for Polyelectrolyte-Contaminated Ion Exchange Resin

- 1) After the cation exchange resin column service ends, perform backwash for 15 minutes. If necessary, inject air to loosen the blocked resin layer.
- 2) Flow 2% NaOH solution at 1BV up-flow through the column for 30 minutes.

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### 2. Contamination of Anion Exchange Resin Columns

#### 2.1 Contamination by Organic Matter

##### 2.1.1 Organic Contamination Mechanism

The process of anion exchange resin becoming organically contaminated involves ion exchange and physical adsorption due to Van der Waals forces. Anion exchange resin not only undergoes ion exchange reactions with inorganic ions during the flow process but also adsorption and exchange reactions with organic matter. Organic matter adsorbed during the service process is desorbed from the resin during the regeneration process using caustic soda, but the desorption rate is slow, and the regeneration process is typically shorter than the service process, leading to accumulation within the resin. The characteristics of organic contamination include long water rinse times, decreased exchange capacity, and particularly for strong base anions, silica leakage.

##### 2.1.2 Regeneration Method for Organic Contaminated Ion Exchange Resin

###### 1) Brine cleaning

- (1) Organic contaminant removal from the resin is conducted at the end of the service process.
- (2) Prepare 3BV of a 10% NaCl brine solution containing 2% NaOH.
- (3) Flow 1BV through the contaminated resin at a speed lower than Specific flowrate 2 BV/h to drain.
- (4) Introduce second 1BV into the resin column and soak for at least 2 hours.
- (5) If possible, stirring the volume by low pressure Air if available. ( 0.3 Bar or so) during the 4) operation can enhance the effect. Drain the second BV.
- (6) Flow the final 1BV through the resin at Specific flowrate 1 BV/h and drain.
- (7) Rinse with pure water.
- (8) Perform double regeneration before returning to service operation.

#### Note

For maximum effectiveness in removing organic contamination, the operational temperature of the brine should be selected as high as possible between a minimum of 35°C and a maximum of 60°C (95 - 140°F).

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### 2.2 Contamination by Silica

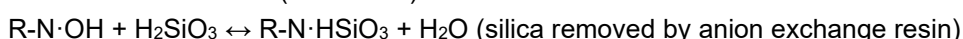
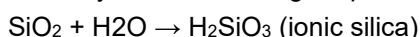
#### 2.2.1 Silica Contamination Mechanism

Natural silicon (Si) mainly exists as various metal silicates or as silica (SiO<sub>2</sub>) in quartz or sand. When surface water comes into contact with silicate minerals, small amounts of silica dissolve, with concentrations varying by region. In water, silica exists in various forms, with the most common being silicate (SiO<sub>3</sub><sup>2-</sup>). In natural water, silicate ions can take many forms, sometimes resembling phosphates, and can agglomerate into colloidal particles large enough to be classified as suspended in solution. At this stage, they are no longer ions and exist in a suspended state in the aqueous solution. This characteristic can pose difficult problems in operation or analysis.

Silica in water can be classified into three categories:

##### 1) Ionic silica

Ionic silica is the part that can be removed by ion exchange resins. Although silica has a very low solubility in water, its solubility increases at higher pH levels.



##### 2) Colloidal silica

It cannot be removed by ion exchange resin. When dried, it loses water and precipitates as SiO<sub>2</sub>. When deposited on an anion exchange resin, it causes performance degradation.

##### 3) Non-ionic silica

It is not ionic and does not form polymers with other ions or substances. It cannot be removed by ion exchange resin and exists in trace amounts.

When regenerating an anion exchange resin, if the temperature is lower than recommended, desorption of silica deposited on the resin is poor, eventually leading to silica contamination. Silica deposited on the anion exchange resin polymerizes and coats the resin surface.

#### 2.2.2 Regeneration Method for Silica-Contaminated Ion Exchange Resin

The silica desorption rate is proportional to the regenerant temperature during regeneration, so it is desirable to heat the regenerant to the highest temperature possible (see table below) without causing performance degradation of the anion exchange resin.

Additionally, brine cleaning may be necessary to remove colloidal silica that is bound to organic matter.

	Ion Exchange Resin	Regenerant Temperature	Remarks
1	Type I Resin	45~50°C	
2	Type II Resin	35~40°C	

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### 3. Others

#### 3.1 Inhibition of Microbial Growth

Normally, when an ion exchange resin demineralization system is used, microbial growth is unlikely due to the significant pH changes during regeneration. However, if the equipment is not operated for a long time, microorganisms can proliferate and contaminate the ion exchange resin. Therefore, in such cases, it is advisable to use a high concentration of NaCl solution to inhibit microbial growth. The procedure is as follows:

- 1) Perform backwash.
- 2) Introduce a 15-20% NaCl solution into the resin column (fill up to the top of the column to minimize the presence of air).
- 3) Perform double regeneration before returning to service operation.

#### 3.2 Contamination by Oil

If the ion exchange resin is contaminated by oil, the anion exchange resin typically has a higher affinity for organic matter, so often only the anion exchange resin is contaminated. Therefore, for mixed bed columns contaminated with oil, it is effective to separate the cation and anion exchange resins and clean only the anion exchange resin. The cleaning procedure for ion exchange resin contaminated by oil is as follows:

- 1) Perform backwash.
- 2) Flow 5BV of 1% H<sub>2</sub>SO<sub>4</sub> or HCl through the resin to convert the anion exchange resin to SO<sub>4</sub> or Cl form. This is done because the anion exchange resin can fracture due to sudden osmotic shock.
- 3) Introduce 1BV of 15% H<sub>2</sub>SO<sub>4</sub> or HCl mixed with 0.2-1.0% non-ionic detergent (which produces less foam) into the resin column.
- 4) Soak for about 4 hours (maintaining a temperature between 40°C and 50°C (104 - 122°F) during this time. Injecting air for about 10 minutes every 2 hours can double the effectiveness).
- 5) Perform backwash for about an hour, soak for another 2 hours, then perform another 30 minutes of backwash.
- 6) Perform double regeneration before returning to service operation.

#### Note

It is advisable to conduct a test in a 500ml beaker before implementing the above procedure on-site. If excessive foam is produced when the detergent is added, it may cause difficulties during on-site implementation

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