Ion Exchange Resins
for Metal Plating and Surface Finishing
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Introduction

Ion exchange resins (IER) can be used in the metal plating and surface finishing industries in a number of ways: rejuvenation of plating baths and pickling solutions, recycling of rinse waters, as well as treatment of waste effluents. Environmental regulations, economic constraints and the quality of the manufactured metal products influence the decision to use ion exchange technology.

Environmental considerations refer to meeting the discharge limits for the various metals imposed by government regulations. These vary from country to country, but in general the limits for discharge of metal finishing effluents into the environment range from 0.1 ppm for the more toxic metals up to 3 ppm for the less toxic metals.

For an economic analysis, the costs of treatment of the plating baths or the rinse waters is compared to the cost of waste discharge. In some cases it is possible to recover and recycle chemicals for their value. Polishing of the effluents is achieved with the use of selective resins, usually of the iminodiacetic acid type, which selectively remove heavy metals from solutions with high Na⁺ and Ca²⁺ backgrounds.

Figure 1 illustrates the overall process of a metal finishing plant. The major sources of the wastes are the spent plating baths and the rinse waters. In many plating industries, different plating baths may be involved. For example, chromium, copper, nickel plating are frequently performed in the same shop. Also, zinc and copper cyanide solutions can be present in the same plant. The wastes from such plating plants are generally treated chemically.

![Figure 1: Overall Process of a Metal Finishing Plant](image-url)
Chromium plating baths are the most common example of a plating bath rejuvenation. A typical spent chromium plating bath contains about 300 g/L of chromic acid and 10-25 g/L of Cu$^{2+}$, Fe$^{3+}$ and Cr$^{3+}$.

The use of a standard cation exchange resin to remove the metal impurities in order to rejuvenate the plating bath is not possible under these conditions. The H$^+$ concentration is so high that the resin will remain in the H$^+$ form, as the equilibrium reaction will shift to the left:

$$\text{RSO}_3^- \text{H}^+ + \text{M}^{2+} \rightleftharpoons (\text{RSO}_3^-)_2 \text{M}^{2+} + \text{H}^+$$

In addition, the high oxidative potential of such a solution may rapidly degrade the resin.

To utilize an IER process, the chromic acid concentration should be reduced to approximately 100 g/L by dilution. A highly crosslinked macroreticular resin such as AMBERLYST 15WET, which has high oxidative stability, may be suitable for this application. Under these conditions, the resin has an operating capacity of about 0.7 eq/L at 3 BV/h flow rate and a regeneration level of 300 g HCl per liter of resin using 10-15% HCl (see figure 2).

Figure 2: Rejuvenation Process for Chromium Plating Bath
Chromic acid baths used for aluminum anodizing are easier to process because they are less concentrated. They contain about 100 g CrO$_3$ /L and 10 g Al$_2$O$_3$ /L as impurities and therefore no dilution is necessary. AMBERLYST 15WET can again be used in this application. This resin can treat about 4 BV of such a solution with less than 1 g Al$_2$O$_3$ /L average leakage. The same flow rates and regenerant levels are used as for chromium plating.

Phosphoric acid solutions can also be rejuvenated with IER. Phosphoric acid is used to passivate and phosphatate iron and steel. These solutions have upper limits for iron content above which the solution becomes exhausted.

A common practice is to remove part of this solution and pass it over a strong acid cation (SAC) resin like AMBERLYST 15WET whereby iron is exchanged for H$^+$ and the purified phosphoric acid returns to the process. Operating conditions are similar as with chromic acid purification described above. The resin can remove up to 35 g Fe$^{2+}$ per liter of resin. Regeneration is done with 2 BV of 10-15% HCl or 10-15% H$_2$SO$_4$. Another case is the purification of HCl pickling solutions, contaminated with Fe$^{2+}$ and Zn$^{2+}$. At these chloride concentrations, Zn$^{2+}$ forms an anionic complex, ZnCl$_4^{2-}$. Iron (Fe$^{2+}$) forms such complexes but at very high HCl concentrations (> 9 N) while Fe$^{3+}$ forms such complexes at about 1.5 N HCl. If both Zn$^{2+}$ and Fe$^{2+}$ are to be removed, Fe$^{2+}$ is first oxidized to Fe$^{3+}$. The HCl is then purified by passing through a strong base anion exchange resin in the chloride form whereby both ZnCl$_4^{2-}$ and FeCl$_4^-$ are exchanged for the Cl$^-$. Regeneration is carried out with water, thereby dropping the HCl concentration inside the resin beads and “breaking” the anionic complexes allowing Zn$^{2+}$ and Fe$^{3+}$ to come off the resin.

Gel type strong base anion (SBA) resins such as AMBERLITE IRA402 Cl, AMBERJET 4200 Cl, AMBERJET 4400 Cl or AMBERLITE IRA400 Cl can be used. The operating capacity depends on both the Fe$^{3+}$ and HCl concentrations. Generally the higher these concentrations, the higher the operating capacity. Operating capacities up to 35 g Fe per liter of resin can be achieved. Flow rates range from about 2-5 BV/h and regeneration is achieved with 4-8 BV of water. Figure 3 illustrates the water elution curves for Fe and Zn using AMBERLITE IRA402 Cl resin.

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**Figure 3: Water Elution of AMBERLITE IRA402 Cl**

Flow rate : 2.5 BV/h

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(1) eq/L$_R$: equivalents per liter of resin

(2) BV: Bed Volume, volume of solution per volume of resin
Due to the use of different plating baths in the same plant, the rinse waters can be acidic (plating baths) or basic (cyanide solutions). The treatment of the rinse waters with IER is performed on the combined rinse waters (caution should be exercised to insure that the pH is not acidic causing formation of HCN), or separately on the acidic and the basic waters.

The concentration of the metal impurities should range at about 1-2 meq/L for this process to be economically feasible. Typically, only the final rinse waters are recycled, as shown in Figure 4.

**Figure 4 : Rinse Water Recycling with IER**
Figure 5 illustrates the recycling of chromic acid plating rinse waters. The composition of such waters is similar to that of the plating bath example given previously, but at a lower concentration. These waters contain around 20 to 100 ppm of chromates with metal impurities (Cu²⁺, Fe³⁺ and Cr³⁺) at 5 to 20 ppm levels. The IER system involves a SAC and a weak base anion (WBA) exchanger. The waste regenerant from the WBA resin, which contains the recovered chromates, is passed through a SAC exchanger in the H⁺ form which converts the chromates to chromic acid. This is subsequently concentrated and recycled back to the plating bath.

A macroreticular SAC resin such as AMBERLYST 16WET or a gel type resin such as AMBERLITE IR120 H can be used. In some installations, AMBERLYST 16WET is preferred due to its macroreticular structure which gives it increased oxidative stability compared to the gel type resins. In addition, better removal of cationic detergents is achieved with this resin.

![Diagram of Acidic Rinse Water Recycling with Chromic Acid Recovery](image-url)

**Figure 5**: Acidic Rinse Water Recycling with Chromic Acid Recovery
Some chromates (Cr\(^{6+}\)) may be reduced on the WBA resin to Cr\(^{3+}\) which can then precipitate on the resin as hydroxides. For that reason, it is recommended to periodically perform an acid clean-up of both the SAC and WBA resins with 15% HCl.

Similar considerations apply for recycling rinse waters from the passivation of steel metals with phosphoric acid. A typical composition of such waters may be 50-100 ppm of PO\(_4^{3-}\) with 5-30 ppm of metal impurities such as Fe\(^{2+}\), Mn\(^{2+}\) and Zn\(^{2+}\). A weak base anion exchange resin such as AMBERLYST A21 can again be used under similar operating conditions and performance, as above.

The basic or mixed rinse waters can contain the following ionic impurities:

<table>
<thead>
<tr>
<th>Cations</th>
<th>Anions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr(^{3+})</td>
<td>CrO(_4^{2-})</td>
</tr>
<tr>
<td>Fe(^{3+})</td>
<td>Fe(II)(CN)(_6^{4-})</td>
</tr>
<tr>
<td>Pb(^{2+})</td>
<td>Fe(III)(CN)(_6^{3-})</td>
</tr>
<tr>
<td>Cu(^{2+})</td>
<td>Cu(I)(CN)(_4^{2-})</td>
</tr>
<tr>
<td>Ni(^{2+})</td>
<td>Ni(CN)(_4^{2-})</td>
</tr>
<tr>
<td>Zn(^{2+})</td>
<td>Zn(CN)(_4^{2-})</td>
</tr>
<tr>
<td>Cd(^{2+})</td>
<td>Cd(CN)(_4^{2-})</td>
</tr>
<tr>
<td></td>
<td>PO(_4^{3-})</td>
</tr>
<tr>
<td></td>
<td>SO(_4^{2-})</td>
</tr>
<tr>
<td></td>
<td>citrates, acetates, tartrates</td>
</tr>
</tbody>
</table>

The flow rate during service cycle can be as high as 20 BV/h. The operating capacity is 0.8 to 1 eq/L\(_R\) when the resin is regenerated with 100 to 120 g HCl per liter of resin using 6-10% HCl in a co-current mode. If H\(_2\)SO\(_4\) is used as regenerant, a 150 g per liter of resin level should be used as a 10% H\(_2\)SO\(_4\) solution. Rinse requirements are about 5 BV. The regeneration level chosen depends on the quality of the recycled water desired. For removal of the strong acids, a macroreticular weak base anion resin such as AMBERLYST A21 is recommended. A typical operating capacity for CrO\(_4^{2-}\) is approximately 1 eq/L\(_R\) when regenerated with 2 to 2.5 eq of NaOH/L\(_R\) (4% NaOH solution). Rinsing is done with 5 BV of decationized water. In general, with the above regeneration levels for the SAC and the WBA resins, a conductivity of about 30 µS/cm is obtained after the WBA resin.
The same SAC and WBA resins can be used in this system as in the preceding one.

Strong base anion resins such as AMBERLYST A26 OH can be used for this application. In the hydroxide form, these resins will remove all weak acids that leak through the WBA resin, such as HCN. Typically an operating capacity of 0.3 to 0.4 eq/L can be achieved using a 100 g NaOH per liter of resin regeneration level (4% NaOH solution). Five BV of deionized water is used for rinsing. A conductivity of about 5 µS/cm is obtained after the SBA resin.

Separate regeneration of the WBA and the SBA resins is recommended in order not to mix the CrO$_4^{2-}$ with the CN$^-$ streams.

Figure 6 illustrates the IER system used to treat these rinse waters. The cationic metal impurities are removed by the SAC resin. The following selectivities of standard gel-type SAC resin are generally observed (3) for a cation with respect to H$^+$:

\[
\begin{align*}
H^+ &< Na^+ < NH_4^+ < Mn^{2+} < K^+ < Mg^{2+} < Fe^{2+} < Zn^{2+} \\
< Co^{2+} < Cu^{2+} < Cd^{2+} < Ni^{2+} < Ca^{2+} < Sr^{2+} < Hg^{2+} \\
< Ag^{+} < Pb^{2+} < Ba^{2+}
\end{align*}
\]

The selectivities of the WBA resin for the anions are as follows:

\[
\begin{align*}
F^- &< oxalate < CF^- < SCN^- < NO_2^- < NO_3^- < SO_4^{2-} \\
< CrO_4^{2-} < Ni(CN)_4^{2-} < Cu(CN)_4^{3-} < Zn(CN)_6^{4-} \\
< Fe(CN)_6^{3-} < OH^-
\end{align*}
\]

Figure 6: Mixed or Alkaline Rinse Waters Recycling

(3) T.V. Arden and F. de Dardel, “Techniques de l’Ingénieur”
Waste Effluent Treatment

Figure 7 illustrates a chemical treatment of galvanic rinse waters. Cr^{6+} (CrO_4^{2-}) is reduced to Cr^{3+} while the cyanides, CN^-, are oxidized to CO_2 and N_2. The IER process is followed by a neutralization and precipitation step where the metallic precipitate is filtered, pressed and disposed as solid wastes. Since the final effluent may still contain some low levels of heavy metals, a polishing unit containing a selective IER such as AMBERLITE IRC748(4) resin is frequently included.

The filtrate after the decantation step contains about 5 to 20 ppm of heavy metals, in addition to higher concentrations of Na^+ and Ca^{2+} salts, in the order of several grams per liter, coming from the neutralizing chemicals.

The stream can be treated by a selective chelating resin, such as AMBERLITE IRC748, usually in a two-columns-in-series, merry-go-round configuration, which can bring the total concentration of metals below 0.1 ppm. AMBERLITE IRC748 has a macroreticular styrene divinylbenzene matrix with iminodiacetic functional groups which can form strong complexes with transition or other metals. The stability of these complexes is in general in the same order as the complexes of the metals with EDTA. For this reason, if the metals are found in the solution as complexes with EDTA, they can not be removed effectively by AMBERLITE IRC748. Similarly, metals such as Zn^{2+}, Ni^{2+} and Cu^{2+} complexed with CN will not be removed by AMBERLITE IRC748. However, in the presence of other complexing agents such as citric acid, AMBERLITE IRC748 efficiently removes metal impurities.

The relative selectivities of several metal ions at a pH of 4 are shown below:

\[
\text{Ca}^{2+} < \text{Mn}^{2+} < \text{Fe}^{2+} < \text{Co}^{2+} < \text{Cd}^{2+} < \text{Zn}^{2+} < \text{Ni}^{2+} < \text{Pb}^{2+} < \text{Cu}^{2+} < \text{Hg}^{2+} < \text{Fe}^{3+}
\]

The affinity for H^+ in the above sequence is situated somewhere between Pb^{2+} and Cu^{2+}. Consequently, for the metals with selectivities less than Cu^{2+}, the resin should be in the salt form (for example in the Na^+ form) otherwise a relatively high metal leakage may be experienced.

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(4) This resin and its use in this type of process are covered under US Patent # 5,804,606, Rohm and Haas Company
Because of the high preference of AMBERLITE IRC748 for the metals, this resin can remove metals from solutions even in the presence of high concentrations of sodium or calcium salts, with very low metal leakage (less than 0.1 ppm with two columns in series). The loading step can be done at a flow rate of 20-30 BV/h while regeneration can be performed at 90 g HCl per liter of resin regeneration level using 5 to 10% HCl, or the equivalent quantity of H$_2$SO$_4$.

After the acid elution, the resin is converted back to the Na$^+$ form. Depending on the NaOH quantity per liter of resin, the conversion to the Na$^+$ form may be total or only partial. The degree of conversion of the resin to the Na$^+$ form as well as the influent composition will affect the effluent pH. In applications involving metal removal, with solutions containing mainly Na$^+$ salts, the resin is converted to about 50% in the Na$^+$ form. In that case, the effluent pH initially is slightly acidic and it becomes progressively neutral as the resin is loaded with the influent Na$^+$ and Ca$^{2+}$.

With solutions containing mainly Ca$^{2+}$ salts, the resin is converted more fully (70% or more) in the Na$^+$ form. In that case, the effluent pH initially is alkaline and becomes progressively neutral as the resin is loaded with the influent hardness ions. In order to more uniformly distribute these 50 or 70% of the sites in the Na$^+$ form, the resin should be conditioned under agitation (see Table below).

### Regeneration Step

<table>
<thead>
<tr>
<th>Direction</th>
<th>Flow rate (BV/h)</th>
<th>Time (minutes)</th>
<th>Quantity (BV)</th>
<th>Level (g/L$_{eq}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rinse</td>
<td>Downflow Water</td>
<td>3</td>
<td>60</td>
<td>3</td>
</tr>
<tr>
<td>Acid regeneration</td>
<td>Downflow 6 % HCl</td>
<td>3</td>
<td>30</td>
<td>1.5</td>
</tr>
<tr>
<td>Rinse</td>
<td>Downflow Water</td>
<td>3</td>
<td>60</td>
<td>3</td>
</tr>
<tr>
<td>Caustic regeneration</td>
<td>4 % NaOH</td>
<td>-</td>
<td></td>
<td>0.7-0.8</td>
</tr>
<tr>
<td>a) 50% Na$^+$ form</td>
<td>Agitation</td>
<td>20</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stand</td>
<td>20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>b) 100% Na$^+$ form</td>
<td>4 % NaOH</td>
<td>3</td>
<td>30-40</td>
<td>1.5-2</td>
</tr>
<tr>
<td>Rinse</td>
<td>Downflow Water</td>
<td>3</td>
<td>60</td>
<td>3</td>
</tr>
</tbody>
</table>
Figure 8 illustrates an example with an influent solution containing 5 ppm of Zn\(^{2+}\), 5 ppm Ni\(^{2+}\) and 2 ppm Cu\(^{2+}\) in CaCl\(_2\) and NaCl background (1 g/L as Ca\(^{2+}\) and 1 g/L as Na\(^+\)) at a pH of 7.

AMBERLITE IRC748 with 70% of the functional groups in the Na\(^+\) form, can treat about 770 BV to a 0.5 ppm Zn\(^{2+}\) end-point at a flow rate of 20 BV/h.

Figure 8: AMBERLITE IRC748 - Typical Leakage Profile
AMBERLITE IRC748 resin has excellent operating capacity for nickel (Ni²⁺), a metal which is more toxic than zinc (Zn²⁺) and for which the tolerated limits in the effluents are lower than those of Zn²⁺.

Another very important feature of AMBERLITE IRC748 is fast kinetics, thus, an increase of the flow rate from 20 to 30 BV/h, results in only a small change in operating capacity, especially if the end of the cycle is fixed at a low metal concentration (Figure 9). The operating conditions for the data in Figure 9 were close to those of Figure 8.
Precious Metal Recovery

The high price of noble or precious metals such as gold, platinum and silver has resulted in the use of IER for recovery from various waste solutions. Gold and silver as cyanide complexes are strongly fixed on SBA exchangers, such as AMBERLITE IRA402 Cl, and are removed quantitatively. In fact, they are so strongly fixed that the resin is dried and incinerated in order to obtain the precious metals. Since this resin can fix up to 150 g Au per liter of resin, the value of the recovered gold represents many times the value of the resin and this procedure is now a common practice.

Silver can be removed from photographic effluents as a thiosulfate complex, \([Ag(S_2O_3)_2]^-\). It can be removed by both SBA resins such as AMBERLITE IRA402 Cl as well as a high capacity acrylic WBA resin such as AMBERLITE IRA67. The elution of Ag from the resin is more efficiently done with ammonium thiosulfate, using 4 BV of 2 M \((NH_4)_2S_2O_3\). The resin therefore works in the \(S_2O_3^-\)\([Ag(S_2O_3)_2]^-\) cycle. The operating capacity of the resin depends on several factors including the ratio of \(S_2O_3^- / [Ag(S_2O_3)_2]^-\) in the influent. Even though initially the operating capacity of AMBERLITE IRA402 Cl may be higher than that of AMBERLITE IRA67, the latter resin regenerates more easily. Operating capacities up to 30 g /L can be obtained, depending on free \(S_2O_3^-\) concentration.

Platinum metals can also be recovered as Cl− complexes on AMBERLITE IRA402 Cl. Thus, Pd, Pt and Ir are removed by AMBERLITE IRA402 Cl and recovered from the resin by incineration. Rh is only fixed weakly and can be eluted with water.

Copper Recovery

Copper can be recovered from acidic solutions and recycled back to the process by using two columns, in series, of AMBERLITE IRC748 with a third column in regeneration (merry-go-round system). AMBERLITE IRC748 can operate in the H⁺ form with no prior conditioning required with NaOH.

With two columns of AMBERLITE IRC748 operating in series at a flow rate of 20 BV/h, the lead column can be run to exhaustion before leakage from the second column exceeds effluent criteria. The third freshly regenerated column can then be put in the polishing position when the lead column is taken off line for regeneration. This operating mode maximizes the capacity of the resin while consistently ensuring that the effluent meets specifications.

Regeneration is performed with 1.5 BV of 10% \(H_2SO_4\) at a flow rate of 2 BV/h. The first half (0.75 BV) of the regenerant contains most of the copper at concentrations around 40 g/L. It is possible to recover copper from this fraction and recycle the second half of the spent regenerant for the next regeneration.

As an illustration, when treating a stream containing 1 g/L of Cu²⁺ (in the form of CuSO₄) at a pH of 3, the lead column will have an operating capacity of approximately 35 g Cu²⁺ per liter of resin in the H⁺ form when Cu²⁺ in the effluent from the lag column reaches a level of 10 ppm. When regenerated as described above, the first half (0.75 BV) of the spent regenerant will contain about 40-45 g Cu²⁺/L, while the second half, containing only minor quantities of Cu²⁺, can be recycled.
Purification of Galvanizing Solutions

The high metal selectivity of AMBERLITE IRC748 especially for Fe$^{3+}$, is used to purify galvanizing solutions from Fe$^{3+}$ impurities. Figure 10 illustrates the Fe$^{3+}$ leakage curve where AMBERLITE IRC748 is used to remove iron impurities from a ZnSO$_4$ galvanizing solution. The composition of this solution was:

<table>
<thead>
<tr>
<th>Ion</th>
<th>Concentration (g/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe$^{2+}$</td>
<td>0.66</td>
</tr>
<tr>
<td>Fe$^{3+}$</td>
<td>0.92</td>
</tr>
<tr>
<td>Zn$^{2+}$</td>
<td>93.5</td>
</tr>
<tr>
<td>Ni$^{2+}$</td>
<td>0.22</td>
</tr>
<tr>
<td>Na$_2$SO$_4$</td>
<td>55.0</td>
</tr>
<tr>
<td>pH</td>
<td>1.72</td>
</tr>
</tbody>
</table>

Fe$^{2+}$ is oxidized to Fe$^{3+}$ using H$_2$O$_2$ and then the solution is passed through AMBERLITE IRC748 in the H$^+$ form at a flow rate of 6.5 BV/h. It should be made sure that there is no excess H$_2$O$_2$ in the solution before passing through the IER in order to avoid resin oxidation. The average iron concentration in the treated solution is decreased by about one half while the resin, even at these highly unfavourable conditions of high ionic background and low pH, removes about 13 g Fe$^{3+}$ per liter of resin.

Regeneration was performed using 2 BV of 15% H$_2$SO$_4$.

Zn/Ni electrolytic solutions can also be treated, where Zn$^{2+}$ and Ni$^{2+}$ concentrations are around 50 g/L each, Na$_2$SO$_4$ around 80 g/L, total iron around 1 g/L and a pH at 1.5-1.9. Operating capacities of 8-10 g Fe per liter of resin can be obtained under these conditions.
## Summary Table of Rohm and Haas Ion Exchange Resins

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Type</th>
<th>Ionic form</th>
<th>Functionality</th>
<th>Application</th>
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<tr>
<td>Amberlite® IR120 H</td>
<td>Gel</td>
<td>Hydrogen</td>
<td>Strong acid</td>
<td>Rinse water recycling</td>
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<td>Amberlyst® 15WET</td>
<td>MR</td>
<td>Hydrogen</td>
<td>Strong acid</td>
<td>Plating bath rejuvenation</td>
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<tr>
<td>Amberlyst® 16WET</td>
<td>MR</td>
<td>Hydrogen</td>
<td>Strong acid</td>
<td>Rinse water recycling</td>
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<tr>
<td>Amberjet® 4200 Cl</td>
<td>Gel</td>
<td>Chloride</td>
<td>Strong base</td>
<td>Plating bath rejuvenation</td>
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<tr>
<td>Amberjet® 4400 Cl</td>
<td>Gel</td>
<td>Chloride</td>
<td>Strong base</td>
<td>Plating bath rejuvenation</td>
</tr>
<tr>
<td>Amberlite® IRA400Cl</td>
<td>Gel, Type I</td>
<td>Chloride</td>
<td>Strong base</td>
<td>HCl pickling solutions</td>
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<tr>
<td>Amberlite® IRA402Cl</td>
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<td>Strong base</td>
<td>HCl pickling solutions</td>
</tr>
<tr>
<td>Amberlyst® A26 OH</td>
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<td>Hydroxide</td>
<td>Strong base</td>
<td>Rinse water recycling</td>
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<td>Amberlite® IRC748</td>
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<td>Weak base</td>
<td>Silver removal</td>
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<td>Weak base</td>
<td>Rinse water recycling</td>
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</table>
Ion Exchange Resins
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Metal01 - December 1999