Dow
Liquid Separations

DOWEX
Ion Exchange Resins

DOWEX ion exchange resins for HFCS deashing and polishing

Technical Manual
DOWEX ion exchange resins for HFCS deashing and polishing

Table of Contents

<table>
<thead>
<tr>
<th>Topic</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn Sweetener Processing</td>
<td>1</td>
</tr>
<tr>
<td>Operating Guidelines</td>
<td>2</td>
</tr>
<tr>
<td>Typical conditions during syrup service</td>
<td>2</td>
</tr>
<tr>
<td>Regenerating deashing resins</td>
<td>2</td>
</tr>
<tr>
<td>Regenerating DOWEX* 88 and DOWEX MONOSPHERE* 88 strong acid cation resins</td>
<td>5</td>
</tr>
<tr>
<td>Regenerating DOWEX 66 and DOWEX MONOSPHERE 77 weak base anion resins</td>
<td>7</td>
</tr>
<tr>
<td>Regenerating mixed bed polishers</td>
<td>9</td>
</tr>
<tr>
<td>Troubleshooting</td>
<td>11</td>
</tr>
<tr>
<td>Troubleshooting deashing systems</td>
<td>12</td>
</tr>
<tr>
<td>Troubleshooting mixed-beds</td>
<td>17</td>
</tr>
<tr>
<td>Resin Properties</td>
<td>18</td>
</tr>
<tr>
<td>Pressure drop as a function of flow rate</td>
<td>19</td>
</tr>
<tr>
<td>Backwash expansion characteristics</td>
<td>21</td>
</tr>
<tr>
<td>Storage and Handling</td>
<td>24</td>
</tr>
<tr>
<td>How to get more information on Dowex products and Dow support services</td>
<td>25</td>
</tr>
<tr>
<td>Tables</td>
<td></td>
</tr>
<tr>
<td>Table 1 Suggested operating conditions for DOWEX deashing and mixed-bed polishing resins</td>
<td>2</td>
</tr>
<tr>
<td>Table 2 Recommended regenerants for DOWEX deashing and mixed-bed polishing resins</td>
<td>6</td>
</tr>
<tr>
<td>Table 3 Recommended quality of regenerants</td>
<td>6</td>
</tr>
<tr>
<td>Table 4 Relative selectivity of DOWEX 88 for cations</td>
<td>7</td>
</tr>
<tr>
<td>Table 5 Typical resin properties for DOWEX deashing and mixed-bed polishing resins</td>
<td>18</td>
</tr>
<tr>
<td>Figures</td>
<td></td>
</tr>
<tr>
<td>Figure 1 Sweetening-off deashing systems</td>
<td>3</td>
</tr>
<tr>
<td>Figure 2 Comparing sweetening-off curves for dextrose deashing</td>
<td>4</td>
</tr>
<tr>
<td>Figure 3 Backwashing deashing resins</td>
<td>4</td>
</tr>
<tr>
<td>Figure 4 Countercurrent vs. cocurrent regeneration</td>
<td>5</td>
</tr>
<tr>
<td>Figure 5 Cation and anion rinses</td>
<td>7</td>
</tr>
<tr>
<td>Figure 6 Weak base anion regeneration and rinsing</td>
<td>8</td>
</tr>
<tr>
<td>Figure 7 Series and recirculation rinsing</td>
<td>8</td>
</tr>
<tr>
<td>Figure 8 Sweetening-off and regeneration of mixed beds</td>
<td>9</td>
</tr>
<tr>
<td>Figure 9 Rinsing and blowdown of mixed beds</td>
<td>10</td>
</tr>
<tr>
<td>Figure 10 Resin mixing in mixed beds</td>
<td>10</td>
</tr>
<tr>
<td>Figure 11 Recycle rinsing of mixed beds</td>
<td>10</td>
</tr>
<tr>
<td>Figure 12 Troubleshooting abrupt deashing problems - Short Cycles</td>
<td>12</td>
</tr>
<tr>
<td>Figure 13 Troubleshooting abrupt deashing problems - Poor Syrup Quality</td>
<td>13</td>
</tr>
<tr>
<td>Figure 14 Troubleshooting abrupt deashing problems - High As-Is Color</td>
<td>14</td>
</tr>
<tr>
<td>Figure 15 Troubleshooting abrupt deashing problems - High After-Heat Color</td>
<td>15</td>
</tr>
<tr>
<td>Figure 16 Troubleshooting abrupt deashing problems - High Pressure Drop</td>
<td>16</td>
</tr>
<tr>
<td>Figure 17 Troubleshooting mixed bed polishers - poor syrup quality</td>
<td>17</td>
</tr>
<tr>
<td>Figure 18 Pressure drop with DOWEX 88 resin</td>
<td>19</td>
</tr>
<tr>
<td>Figure 19 Pressure drop with DOWEX 66 resin</td>
<td>19</td>
</tr>
<tr>
<td>Figure 20 Pressure drop with DOWEX MONOSPHERE 88 and MONOSPHERE 77 resins</td>
<td>19</td>
</tr>
<tr>
<td>Figure 21 Pressure drop with DOWEX 88 MB resin</td>
<td>20</td>
</tr>
<tr>
<td>Figure 22 Pressure drop with DOWEX 22 resin</td>
<td>20</td>
</tr>
<tr>
<td>Figure 23 Backwash expansion of DOWEX 88 resin</td>
<td>21</td>
</tr>
<tr>
<td>Figure 24 Backwash expansion of DOWEX MONOSPHERE 88 resin</td>
<td>21</td>
</tr>
<tr>
<td>Figure 25 Backwash expansion of DOWEX 66 resin</td>
<td>21</td>
</tr>
<tr>
<td>Figure 26 Backwash expansion of DOWEX MONOSPHERE 77 resin</td>
<td>22</td>
</tr>
<tr>
<td>Figure 27 Backwash expansion of DOWEX 88 MB resin</td>
<td>22</td>
</tr>
<tr>
<td>Figure 28 Backwash expansion of DOWEX 22 resin</td>
<td>23</td>
</tr>
</tbody>
</table>

*Trademark of The Dow Chemical Company
The use of DOWEX ion exchange resins in corn sweetener processing

Millhouse → Starch Slurry → Gelatinization → Dextrinization → Saccharification

吸附剂脱色
DOWEX* OPTIPORE* SD-2 吸附剂

麦芽糖侧脱脂
DOWEX MONOSPHERE* 88
正离子树脂

DOWEX MONOSPHERE 77
负离子树脂

蒸发 (某些系统)

糖化

Dextrose Side Deashing
DOWEX MONOSPHERE 77
Anion Resin

Evaporation (some systems)

Isomerization

Fruuctose Side Deashing
DOWEX MONOSPHERE 88
Cation Resin

吸附剂脱色
DOWEX OPTIPORE SD-2 吸附剂

Evaporation

42% 麦芽糖产品

混合床抛光
DOWEX 22
负离子树脂
DOWEX 88MB
正离子树脂

分离
DOWEX MONOSPHERE 99 树脂

80-90% 麦芽糖 → 混合

80-90% 葡萄糖

Raffinate (Recycled in Process)

DOWEX MONOSPHERE 77
Anion Resin

DOWEX 88MB
Cation Resin

蒸发

55% HFCS 产品

Vacuum Filtration or Membrane Separation of Insolubles

真空过滤或膜分离不溶物
Operating Guidelines

Typical conditions during syrup service

To obtain optimum performance and long life from DOWEX ion exchange resins, the conditions under which they operate must be maintained within certain parameters. In particular, DOWEX products vary in their temperature sensitivity. Table 1 provides data on suggested operating conditions such as maximum syrup temperatures.

When to regenerate deashing resins

In a double pass system, when the primary deashing unit becomes exhausted to the point where syrup quality drops below an acceptable level, it is taken off-line and replaced with the secondary unit. An off-line, regenerated unit then becomes the secondary unit. This point, called breakthrough, is determined by measuring the conductivity and/or the pH of the syrup as it leaves the primary deashing anion unit. When the conductivity increases to around 20-30 micromhos per centimeter or the pH drops to around 4.5, it is generally time to regenerate the unit.

Regenerating deashing resins

While specific configurations of deashing units vary in the industry, the basic principles involved in regeneration are relatively standard. This section offers a broad overview of the process. Specific procedures may vary, and additional steps may be required, depending on the design and operation of your system.

Table 1 – Suggested operating conditions for DOWEX deashing and mixed-bed polishing resins

<table>
<thead>
<tr>
<th></th>
<th>DOWEX 88 strong acid cation</th>
<th>DOWEX MONOSPHERE 88 strong acid cation</th>
<th>DOWEX 66 weak base anion</th>
<th>DOWEX MONOSPHERE 77 weak base anion</th>
<th>DOWEX 88 MB strong acid cation</th>
<th>DOWEX 22 strong base anion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum syrup</td>
<td>200° F 93°C (H+ form)</td>
<td>200° F 93°C (H+ form)</td>
<td>140° F 60°C (FB form)</td>
<td>140° F 60°C (FB form)</td>
<td>200° F 93°C (H+ form)</td>
<td>115° F 46°C (OH- form)</td>
</tr>
<tr>
<td>Temperature</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bed Depth, (minimum)</td>
<td>36 inches 91 cm</td>
<td>36 inches 91 cm</td>
<td>36 inches 91 cm</td>
<td>36 inches 91 cm</td>
<td>36 inches 91 cm</td>
<td>36 inches 91 cm</td>
</tr>
<tr>
<td>Regenerant Level</td>
<td>6-7 lbs/cu. ft. 96-112 kg/m³</td>
<td>5-6 lbs/cu. ft. 80-96 kg/m³</td>
<td>5-6 lbs/cu. ft. 80-96 kg/m³</td>
<td>4-5 lbs/cu. ft. 64-80 kg/m³</td>
<td>6-7 lbs/cu. ft. 96-112 kg/m³</td>
<td>4-5 lbs/cu. ft. 64-80 kg/m³</td>
</tr>
<tr>
<td>(100% basis)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Regenerant</td>
<td>7% HCl</td>
<td>7% HCl</td>
<td>4% NaOH</td>
<td>4% NaOH</td>
<td>7% HCl</td>
<td>4% NaOH</td>
</tr>
<tr>
<td>Concentration</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Regenerant</td>
<td>200°F 93°C</td>
<td>200°F 93°C</td>
<td>140°F 60°C</td>
<td>140°F 60°C</td>
<td>200°F 93°C</td>
<td>115°F 46°C</td>
</tr>
<tr>
<td>Temperature (max.)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Substitute</td>
<td>5% Na₂CO₃ @ 7-8 lbs/ft³ (112-128 kg/m³)</td>
<td>5% Na₂CO₃ @ 6-7 lbs/ft³ (96-112 kg/m³)</td>
<td>5% Na₂CO₃ @ 6-7 lbs/ft³ (96-112 kg/m³)</td>
<td>7% Na₂CO₃ @ 5-6 lbs/ft³ (80-96 kg/m³)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Regenerants</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>5% NH₄OH @ 5-6 lbs/ft³ (80-96 kg/m³)</td>
<td>5% NH₄OH @ 4-5 lbs/ft³ (64-80 kg/m³)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Never use oxidizing agents such as nitric acid, perchlorates, or hydrogen peroxide with ion exchange resins. The reaction can cause slight to severe degradation of the resin, possibly producing explosive reaction products. Also, the use of H₂SO₄ to regenerate cation resins is typically discouraged because CaSO₄ can precipitate in the ion exchange resin.

Since the performance of ion exchange resins is dependent on proper regeneration, it is important to closely monitor your regeneration procedures. For example, routinely have your quality assurance lab check regenerant quality and concentration. Also be sure that meters, pumps, and valves are working and are maintained properly.

**Sweetening-off**

When the on-line deashing unit reaches breakthrough, the syrup feed is discontinued and water (generally at the process flow rate) is used to push the syrup off the resin bed (Figure 1). The full strength syrup exiting the bed during the early stage of sweetening-off goes forward in the process. On the dextrose side, when sweetwater (diluted syrup) starts to exit the bed, it can often be put back into the process. With fructose side deashing, however, sweetwater is not generally recycled because it contaminates the glucose stream with fructose. When the dissolved solids concentration of the syrup gets down to a fraction of a percent, the effluent is switched to waste. The use of deionized (condensate) water is not essential for sweetening-off; however, hard (raw) water will further exhaust the resins. A rule of thumb for water usage is to use the water of highest conductivity for sweetening off and save the best water for regeneration final rinse.

Due to better resin kinetics, DOWEX MONOSPHERE ion exchange resins sweeten-off more efficiently, resulting in 30-40% less sweetwater and 40-60% less wastewater generated per cycle on both the dextrose and fructose sides. Figure 2 shows the shorter, steeper sweetening-off profile of a DOWEX MONOSPHERE 88 and 66 resin pair compared with conventional deashing resin pair.

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**Figure 1 – Sweetening-off deashing systems**

![Diagram of sweetening-off deashing systems](image-url)
**Backwashing**

The next step is backwashing, fluidizing the bed by pumping water upflow. By lifting and separating the beads, backwashing aids in thorough cleaning of the bed and also allows the beads to reclassify in the bed, improving flow distribution. Backwashing removes residual syrup, resin fines, microorganisms, and other matter to allow good regenerant contact and flow through the bed (Figure 3). A minimum 50% expansion of the bed volume during backwashing is recommended; 100% expansion is even better.

Unscreened backwash outlets are most effective because they allow the contaminants to freely exit the bed; however, backwash expansion must be monitored carefully to ensure that resin beads don’t escape the bed. This is particularly true with anion resins, which are less dense than cation resins.

Problems sometimes occur when the water temperature is lower than normal because colder water will expand the resin bed more at a given flow rate. Flow rates should be decreased when using colder water. Backwash expansion curves for DOWEX resins are provided on pages 21-23 to help you determine the expansion of your beds at a given temperature and flow rate.

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**Figure 2 – Comparing sweetening-off curves for dextrose deashing**

This graph tracks the dextrose side cation-anion effluent during sweetening-off. Notice the shorter, steeper profile of DOWEX MONOSPHERE resins. More efficient rinsing and longer service cycles each lead to significant reductions in sweetwater and wastewater.

**Figure 3 – Backwashing deashing resins**

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Regenerating DOWEX 88 and DOWEX MONOSPHERE 88 strong acid cation resins

Two methods of regenerating are in common use: cocurrent (in the same direction as syrup flow) and countercurrent (opposite syrup flow). We recommend countercurrent cation regeneration at normal regenerant loads, particularly for single pass systems, because it results in lower sodium leakage when the unit is returned to service.

Since 100% regeneration of the resin is not economical, a small percentage of exchange sites will still be occupied by salts. With cocurrent regeneration, these residual salts (called the salt heel) end up at the bottom of the bed and can result in higher than acceptable sodium leakage when the bed is returned to syrup service (Figure 4).

With countercurrent regeneration, the salt heel ends up at the top of the bed, and even if the syrup picks up some of this salt, it will be removed by the more fully regenerated resin lower in the bed. Salt leakage due to cocurrent regeneration is more pronounced in single pass systems than in double pass systems (at equivalent acid loads).

With both methods of regeneration a build-up of calcium and/or magnesium may occur on the resin over several cycles. This may require extra heavy acid dosages on a periodic basis in order to maintain normal operating capacities.

Cation resin regeneration efficiency

Prudent operation of ion exchange systems is a trade-off between apparent short-term savings and long-term operating costs. The most important factor in cation resin regeneration efficiency is the acid concentration. Since the resin's active sites have a greater affinity for the salts they have picked up than for hydrogen ions, a sufficient acid concentration is required to overwhelm and drive the salts off these sites. Technically, this is called mass action. Even though 40% over the stoichiometric amount of acid will not completely regenerate the resin, the use of additional excess acid is not justified by the economics.

The standard recommendation for regenerating DOWEX 88 strong acid cation resins is 7% hydrochloric acid (2N) at 6-7 pounds of 100% HCl per cubic foot of resin.¹ These conditions have proven to be the most efficient and economic for routine regeneration in most systems. Higher concentrations or loads will regenerate the resins more completely, but the minimal capacity gained is generally not worth the extra cost in acid. Lower concentrations or loads will result in inefficient regeneration of the ion exchange resins' capacity and reduced lifetime due to irreversible accumulation of impurities.

Acid contact time is also important to regeneration efficiency. We recommend a minimum of 45 minutes acid pumping time to allow mass action to take place.

Regeneration efficiency is also dependent on the purity of the acid and dilution water. Table 3 gives the minimum purity requirements for regenerants commonly used with DOWEX resins.

Regeneration efficiency is reduced by increased amounts of calcium and magnesium loaded on the resin because of the high selectivity of the cation resin for these salts (Table 4). Extra acid (120-140% of the recommended load) may be required to displace these salts.

Bead size also affects the regeneration efficiency. Larger beads require longer acid contact time for complete regeneration than smaller beads. Because they permit a smaller average size bead to be used without excessive pressure drop, DOWEX MONOSPHERE resins regenerate more efficiently than standard DOWEX resins. This can result in higher operating capacity and 15-20% longer service cycles. Longer cycles translate into fewer regenerations in a given time span, significantly reducing regenerant costs and increasing resin lifetimes.

¹For DOWEX MONOSPHERE 88 resin, the recommended load drops to 5-6 pounds per cubic foot.
**Table 2 – Recommended regenerants for DOWEX deashing and mixed-bed polishing resins**

<table>
<thead>
<tr>
<th>Regenerant level(^1) (100% basis)</th>
<th>DOWEX 88 strong acid cation</th>
<th>DOWEX MONOSPHERE 88 strong acid cation</th>
<th>DOWEX 66 weak base anion</th>
<th>DOWEX MONOSPHERE 77 weak base anion</th>
<th>DOWEX 88 MB strong acid cation</th>
<th>DOWEX 22 strong base anion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regenerant concentration (minimum)</td>
<td>6-7 lbs. HCl/cu. ft.</td>
<td>5-6 lbs. HCl/cu. ft.</td>
<td>5-6 lbs. NaOH/cu. ft.</td>
<td>4-5 lbs. (64-80 kg/m(^3)) NaOH/cu. ft.</td>
<td>6-7 lbs./cu. ft. HCl</td>
<td>4-5 lbs. NaOH/cu. ft.</td>
</tr>
<tr>
<td></td>
<td>96-112 kg/m(^3)</td>
<td>80-96 kg/m(^3)</td>
<td>7-8 lbs. (112-128 kg/m(^3)) Na(_2)CO(_3)/cu. ft.</td>
<td>6-7 lbs. Na(_2)CO(_3)/cu. ft.</td>
<td>96-112 kg/m(^3) Na(_2)CO(_3)/cu. ft.</td>
<td></td>
</tr>
<tr>
<td>Regenerant temperature (max)</td>
<td>7% HCl</td>
<td>7% HCl</td>
<td>4% NaOH</td>
<td>4% NaOH 5% Na(_2)CO(_3) 5% NH(_4)OH</td>
<td>7% HCl</td>
<td>4% NaOH 7% Na(_2)CO(_3)</td>
</tr>
<tr>
<td></td>
<td>200° F 93°C</td>
<td>200° F 93°C</td>
<td>140°F 60°C</td>
<td>140°F 60°C</td>
<td>200° F 93°C</td>
<td>115°F 46°C</td>
</tr>
</tbody>
</table>

\(^1\)Assuming a minimum 90% equipment efficiency.

**Table 3 – Recommended quality of regenerants**

<table>
<thead>
<tr>
<th>Caustic Soda (NaOH)</th>
<th>Hydrochloric Acid (HCl)</th>
<th>Ammonium Hydroxide (NH(_4)OH)</th>
<th>Soda Ash (Na(_2)CO(_3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Basis</td>
<td>Grade: Technical</td>
<td>Liquid ammonia gassified and dissolved in water is generally pure enough for regeneration of weak base anion resins.</td>
<td>Grade: Technical, white powder</td>
</tr>
<tr>
<td>&lt;1200 ppm NaCl</td>
<td>28% (18° Be(^{\prime})) HCl</td>
<td></td>
<td>Typical analysis: 99% Na(_2)CO(_3), 2100 ppm NaCl</td>
</tr>
<tr>
<td>&lt;3000 ppm Na(_2)CO(_3)</td>
<td>&lt;100 ppm Fe</td>
<td></td>
<td>200 ppm Na(_2)SO(_4)</td>
</tr>
<tr>
<td>&lt;30 ppm NaClO(_3)</td>
<td>&lt;100 ppm Fe</td>
<td></td>
<td>22 ppm Fe(_2)O(_3)</td>
</tr>
<tr>
<td>&lt;10 ppm Fe</td>
<td>&lt;5 ppm oxidants as Cl(_2)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>&lt;2000 ppm Na(_2)SO(_4)</td>
<td>&lt;4000 ppm sulfate</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 4 – Relative selectivity of DOWEX 88 for cations

<table>
<thead>
<tr>
<th>Ion</th>
<th>Selectivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>H⁺</td>
<td>1</td>
</tr>
<tr>
<td>Na⁺</td>
<td>2</td>
</tr>
<tr>
<td>K⁺</td>
<td>3</td>
</tr>
<tr>
<td>Mg²⁺</td>
<td>3</td>
</tr>
<tr>
<td>Ca²⁺</td>
<td>6</td>
</tr>
</tbody>
</table>

The fact that Na⁺, K⁺, Mg²⁺, and Ca²⁺ all have a higher affinity for the resins’ active sites than the hydrogen ion is the basis for the cation resins’ ability to effectively remove unwanted salts from the syrup stream. Because regeneration must overcome these selectivity ratios, the concentration and contact time of the regenerant must be sufficient to overwhelm the sites with hydrogen ions and force these salts off.

**Cation resin rinsing**

Following regeneration are two rinse steps: a slow rinse and a fast rinse (Figure 5). The slow rinse is performed in the same direction as the regenerant flow. The purpose of the slow rinse step is to extend the contact time of the acid. This time extension improves regeneration efficiency and allows the displaced salts time to diffuse out of the interior of the resin beads and into the rinse stream. Slow rinse is performed at regeneration flow rates using condensate or deionized water. The slow rinse is typically continued until there is a noticeable acid dilution (the pH rises).

Following the slow rinse, a fast rinse at 2 to 4 times the slow rinse flow rate is performed to wash the residual, dilute acid off the resin. This rinse is continued until the effluent quality reaches the desired level, typically 3-5 pH. The water used for this rinse must be of especially high quality (condensate or deionized water). Poor quality rinse water will partially exhaust the regenerated resin before the resin is even returned to syrup service. If the cation resin has been kept reasonably clean, part of the rinse water can usually be recovered for subsequent acid dilution and/or rinse water.

**Regenerating DOWEX 66 and DOWEX MONOSPHERE 77 weak base anion resins**

In regenerating DOWEX 66 and DOWEX MONOSPHERE 77 resins, the objective is to remove the acids picked up during syrup service (i.e., sulfuric, nitric, hydrochloric, and organic acids). Regeneration is almost always done downflow (Figure 6). With the proper regenerant concentration and good flow distribution, weak base anion resins can be regenerated with nearly 100% efficiency. The minimum regeneration recommendations are 4% sodium hydroxide at 5-6 pounds per cubic foot for DOWEX 66 and 4-5 pounds per cubic foot for DOWEX MONOSPHERE 77. Alternatively, 5% soda ash or 5% ammonium hydroxide can be used as specified in Table 2. As with cation resin regeneration, 45 minutes pumping time is recommended as a minimum.

**Rinsing weak base anion resins**

The slow and fast steps of rinsing anion resins are generally performed in the same manner as previously described for cation resins. The slow rinse is performed at the regeneration flow rate until noticeable dilution of the regenerant at the discharge. The subsequent fast rinse is continued until the discharge conductivity or pH drops below the syrup cycle breakthrough point.

Rinse requirements for weak base anion resins increase over time as the resin progressively fouls. Ammonium hydroxide-regenerated resins give the lowest rinse requirements. On the other hand, ammonium hydroxide-regenerated resins tend to foul out more rapidly.

**Series and recirculation rinsing**

Series rinsing of cation and anion beds can be used to conserve rinse water (Figure 7). Series rinsing can also provide deionized water for subsequent rinses and dilutions. Series rinsing involves pumping rinse water through the cation and the anion beds in series.

Recirculation rinsing is sometimes performed when rinse requirements become excessive (i.e., older resins) and continuous pumping of new rinse water isn’t economically justified. Recirculation rinsing involves pumping rinse water through the cation and anion beds in a closed loop. During this process, the residual acid coming off the cation bed is removed by the anion resin. At the same time, the residual caustic com-
ing off the anion bed is neutralized when returned to the cation bed.
Recirculation rinsing helps to achieve low conductivity effluent during the sweetening-on step. It can also help reduce salt leakage during the syrup cycle. However, recirculation rinsing consumes a small amount of the cation and anion resin capacities.

**Sweetening-on deashing systems**
The sweetening-on procedure is essentially the opposite of the sweetening-off procedure. When the on-line unit pair in the primary position reaches breakthrough, the regenerated unit pair is switched to syrup service. For a double-pass system, the secondary unit pair is moved into the primary position and the fresh unit is brought into the secondary position. Thus, sweetening-on is accomplished in-line, at the process flow rate. The effluent from the fresh unit pair is typically handled as treated water and waste. Next comes sweetwater. Finally, when the syrup concentration is high enough, the treated syrup is sent forward in the process. At the same time the fresh unit pair is sweetening-on, the exhausted unit pair is sweetening-off.

**Cross-regenerating deashing resins**
Weak base anion resins generally require cross-regeneration with 7% HCl and 4% NaOH approximately every 6 weeks, on average. Cross-regeneration helps cleanup organic fouling and extends the life of the resin.

**Caustic brine cleaning of anion resins**
For highly fouled resin, soaking the resin in a 2% caustic soda/10% sodium chloride brine solution will help restore the resin’s capacity. This treatment may be done every 6 months or so to keep the resin in good condition. Caustic brine cleaning is recommend-ed only when the cleaning has been done at regular intervals starting when the resin was new.

---

**Figure 6 – Weak base anion regeneration and rinsing**

![Diagram](image.png)

Series and recirculation rinsing are techniques to minimize water use.

**Figure 7 – Series and recirculation rinsing**

![Diagram](image.png)

We also recommend cross-regeneration of strong acid cation resins periodically, using caustic soda, rinsing, then regenerating with hydrochloric acid.
Regenerating Mixed Bed Polishers

Regeneration of mixed bed polishers is more complicated than regeneration of split beds because the anion and cation resins are intimately mixed during syrup service. As part of the regeneration the cation and anion beads must be separated prior to regenerant chemical contact. For quality guidelines, refer to Table 3, Page 6.

Sweetening-off and backwashing

The first step in regeneration of mixed bed polishers (Figure 8) is sweetening-off in essentially the same manner previously described for deashing units.

Next, the resins are backwashed. Backwashing causes the denser, larger cation resin beads to migrate to the lower portion of the expanded bed, while the anion resin beads rise to the top. After backwashing, the bed is allowed to settle, resulting in two distinct layers.

Chemical addition

The resins are regenerated by pumping caustic soda or sodium carbonate through the anion resin from the top of the bed while pumping hydrochloric acid through the cation resin from the bottom. Excess regenerants meet at the central lateral, neutralize each other, and are sent to waste. Since this procedure requires that the interface of the two resins occurs precisely at the same level as the lateral discharge, it is critical that the correct cation resin volume is maintained in the bed.

The minimum recommendation for regeneration of DOWEX 88MB strong acid cation resin is 6-7 lbs/cu ft of 7% hydrochloric acid.

For DOWEX 22 strong base anion resin, the minimum recommendation is 4% sodium hydroxide at 4.5 lbs/cu. ft. or 7% soda ash at 5-6 lb/cu. ft.

Rinsing and blowdown of mixed beds

Following regeneration, a slow rinse is performed maintaining the same flow directions as the regenerants (Figure 9). Next, a fast rinse is performed from the top and bottom of the bed simultaneously. Both rinses require demineralized or deionized water. A blowdown of the liquid head to just above the resin level is typically performed after rinsing to accommodate the subsequent mixing step.

Figure 8 – Sweetening-off and regeneration of mixed beds

[Diagram showing water, process, sweet water, waste, full strength, cation, anion, and waste streams during regeneration process]
**Remixing mixed beds for syrup service**

Complete and intimate mixing of the cation and anion resins is essential for proper operation of mixed beds. Typically, the resin bed is first expanded using air and water simultaneously to mix the resins (Figure 10). Once the resins are intimately mixed, water addition is stopped but air continues to be blown into the bed until the bed can finish settling without significant separation of the anion resin from the mixture. The system is vented, and the bed starts to settle. As it settles, water is drained off at a rate which keeps the water level just above the top of the resins. This keeps the anion resins from separating near the top of the bed. Usually this sequence is part of the automatic operating program, but in some systems it is performed manually.

**Recycle rinsing of mixed beds**

A recycle rinse is also commonly used with mixed beds because it helps achieve a low conductivity effluent during sweetening-on and syrup service (Figure 11). Because the performance requirements of mixed bed polishers are more stringent than with deashing beds, effluent rinse water conductivity should ideally be below 10 micromhos/cm near the end of the rinse. At the completion of the recycle rinse a blowdown of the liquid head is used to remove the water to just above the resin level in systems operated with air domes.
Troubleshooting
deathing systems

Abrupt vs. gradual problems

The following troubleshooting flow charts will help you determine the cause of problems which occur abruptly in deashing and mixed-bed polishing units. These charts also suggest corrective action. Abrupt problems are those which occur within a few minutes, hours, days, or even weeks. These problems are distinguished from the gradual decrease in unit performance which results from normal aging and occurs over much longer periods of time (months and years).

The value of routine analysis and good record-keeping

When problems do occur, the task of troubleshooting will be greatly simplified if you regularly sample and analyze your resins and keep good records on your system. This will allow you to compare current performance with normal operation to determine the extent and sometimes the cause of the problem. In fact, many potential problems can be identified through routine resin analysis before they show up in the form of short cycles or poor syrup quality. That’s why we encourage processors to take advantage of our Resin Check-Up Service.

Our Resin Check-Up Service helps you obtain optimum results from DOWEX resins

This analytical service covers every critical operating characteristic of your resin. These analyses allow us to help you maximize the remaining usable life of the resin. Each time you send samples to our lab you’ll receive a complete report which includes itemized listings of the operating characteristics as well as recommendations for remedial steps, if required. We also maintain a historical database on your resins which can prove extremely valuable in predicting or troubleshooting possible problems.

Our lab offers one of the most complete analytical services available for producers of nutritive sweeteners.

In addition to the standard tests, we have the capability of running a wide variety of non-standard tests to assist you in troubleshooting your system. Syrup samples can also be evaluated for resin-related quality problems.

System profiling helps you fine-tune your system

System profiling is another service available to users of DOWEX resins. We start by taking syrup samples at various points in the system over a complete cycle. The special battery of tests we perform on these samples gives us the information we need to help you fine-tune your system for economical operation and consistently high syrup quality.
First determine if the problem is coming from the cation bed or the anion bed by sampling the anion discharge at service breakthrough.

- **Symptoms**
  - **Breakthrough on Cation Unit**
    - If pH is greater than 4 and there is high conductivity, then salt breakthrough is probably occurring on the cation unit.
  - **Breakthrough on Anion Unit**
    - If pH is below 4, then acid breakthrough is probably occurring on the anion unit.

- **Possible Causes & Suggested Actions**
  - **Poor Cation Regeneration**
    - Check acid strength, load, quality, and flow rates (regenerant dilution and contact time). Check rinse water and dilution water quality. See Table 3, page (6).
  - **High Feed Ash**
    - Check for excess enzyme activator addition or excessive pH adjustment upstream. Check if feed syrup or anion regenerant is leaking to product line.
  - **Poor Flow Distribution**
    - Check for mechanical problems that could affect flow during regeneration or syrup service such as leaky valves or broken distribution laterals.
  - **Poor Anion Regeneration**
    - Check base strength, load, quality, and flow rates (regenerant dilution and contact time). Check rinse water and dilution water quality. See Table 3, page (6).
  - **High Feed Ash**
    - Check for excess enzyme activator addition or excessive pH adjustment upstream. Check if cation regenerant (acid) is leaking to product line.
  - **High Organic Acids & Microbial Contamination**
    - Check for microbe buildup ahead of and on anion bed. If found, backwash around screening devices; clean up and regenerate cation and anion beds; clean up surge tanks; heat exchangers, etc.
If pH is below normal, the problem is generally with the anion unit. If low, top off with new resins.

If pH is equal to or greater than normal and there is high conductivity, the problem is generally with the cation unit. Check acid strength, load, quality, and flow rates (regenerant dilution and contact time). Check rinse water and dilution water quality - If OK, increase acid dosage.

pH ≥ normal

Symptoms

Leakage from Cation Unit
If pH is equal to or greater than normal and there is high conductivity, the problem is generally with the cation unit.

Leakage from Anion Unit
If pH is below normal, the problem is generally with the anion unit.

pH < normal

Possible Causes & Suggested Actions

Poor Cation Regeneration
Check acid strength, load, quality, and flow rates (regenerant dilution and contact time). Check rinse water and dilution water quality - If OK, increase acid dosage.

Poor Flow Distribution
Check for mechanical problems that could affect flow during regeneration or syrup service such as leaky valves or broken distribution laterals - If OK, increase acid dosage.

Regenerant Leakage to Line
Alkali regenerant leaking to the product line will cause a high pH that has nothing to do with the cation bed. Check for leaking valves.

Regenerant Leakage to Line
Acid regenerant leaking to the product line will cause a low pH that has nothing to do with the anion bed. Check for leaking valves.

High Organic Acids & Microbial Contamination
Check for microbe buildup ahead of and on anion bed. If found, backwash around screening devices; clean up and regenerate cation and anion beds; clean up surge tanks, heat exchangers, etc.
Figure 14: Troubleshooting abrupt deashing problems - High As-is Color

Problem | Possible Causes | Suggested Actions
--- | --- | ---
Color (operational) | Poor Operation of Cation Unit | See Figure 13, "Troubleshooting Poor Syrup Quality" (cation branch)
| Poor Operation of Anion Unit | See Figure 13, Troubleshooting Poor Syrup Quality (anion Branch)
| High Temperature | Reduce Temperature or Increase Flow Rate
| Long Retention Time | Reduce Time or Decrease Recycle

Pink Color (high pH environment) | Weak Base Anion Resin with Excessive Salt Splitting Capacity | Call your Dow Technical Service Representative for Assistance
| Poor Cation Performance with New Anion Resin | See Figure 13, "Troubleshooting Poor Syrup Quality" (cation branch).
| Caustic in Vessel and/or Lines | Replace Cation Resin
| | Check Rinse Effectiveness
| | Check for Anion Regenerant Leakage
Figure 15: Troubleshooting abrupt deashing problems - High After-Heat Color

Problem: High After-Heat (heat treated) Color

Possible Causes: Protein Leakage from Cation Unit

Suggested Actions:
- See Figure 13, "Poor Syrup Quality" Follow Cation Unit branch of chart (pH ≥ normal).
- Check for Increases in Incoming Protein Check for enzymes or corn protein.
**Possible Causes**

**Defective Operating Devices**
Plugged valves, screens, and distribution laterals are the most common cause of abrupt pressure drop.

**Microbial Contamination**
Pressure drop due to microbial buildup generally builds up gradually.

**Resin Breakage**
If resin breaks up rapidly, something is wrong in the system that needs to be corrected. For example, an oxidizer such as oxygen or chlorine may be entering the system.

**An Abrupt Rise In Pressure Drop**
This is usually due to a defective operating device. Typically, problems caused by contamination or resin breakage will gradually appear over a period of weeks or months.

**Suggested Actions**

**Check for Plugging**
Check line valving, strainers, and distribution laterals for mechanical problems or plugging that can inhibit flow.

**Remove Cell Masses**
Remove cell masses from the top of resin beds by backwashing (bypassing screen devices), and/or mechanical removal (skimming bed tops).

**Increase Service Temperature**
Increase the ion exchange system service temperature to 46°C (115°F).

**Add SO₂ to Feed**
Add 100 ppm SO₂ to ion exchange feed on periodic basis to minimize microbial activity and block browning reactions.

**Terminate Runs on Color**
Terminate extra long runs on color rather than conductivity or pH.

**Check For Resin Fines**
Sample the resin bed and check for fines after the regeneration cleanup sequence. Remove fines by backwashing, bypassing screening devices.
Troubleshooting Mixed-beds

Figure 17: Troubleshooting abrupt problems - Poor Syrup Quality

Cation or Anion Problem?
First determine if the problem is in the cation or the anion resin by checking the syrup quality at the discharge from the unit.

If pH is Greater than Normal and/or Cation Leakage -
The problem is generally with the cation resin.

- Insufficient Cation Resin Regeneration
  Check regenerant strength, quality, and flow rates. Check rinse water quality.

- Regenerant Cross-Contamination
  If the cation/anion resin interface is high, the anion regenerant will contaminate the top portion of the cation resin, putting it into the sodium form and causing salt leakage.

If pH is Less than Normal (4-6) and/or Anion Leakage -
The problem is generally with the anion resin.

- Insufficient Anion Resin Regeneration
  Check regenerant strength, quality, and flow rates. Check rinse water quality.

- Regenerant Cross-Contamination
  If the cation/anion resin interface is too low, the cation regenerant will contaminate the lower portion of the anion resin, causing chloride leakage when the unit is returned to service.
Resin Properties

This section provides typical resin properties as well as pressure drop and backwash expansion charts for DOWEX resins.

Table 5 – Typical resin properties for DOWEX deashing and mixed-bed polishing resins

<table>
<thead>
<tr>
<th></th>
<th>DOWEX 88</th>
<th>DOWEX MONOSPHERE 88</th>
<th>DOWEX 66</th>
<th>DOWEX MONOSPHERE 77</th>
<th>DOWEX 88 MB</th>
<th>DOWEX 22</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type</td>
<td>Strong acid cation</td>
<td>Strong acid cation</td>
<td>Weak base anion</td>
<td>Weak base anion</td>
<td>Strong acid cation</td>
<td>Strong base anion, Type II</td>
</tr>
<tr>
<td>Active group</td>
<td>Sulfonate</td>
<td>Sulfonate</td>
<td>Tertiary amine</td>
<td>Tertiary amine</td>
<td>Sulfonate</td>
<td>Quaternary amine</td>
</tr>
<tr>
<td>Ionic form (as produced)</td>
<td>Sodium</td>
<td>Sodium</td>
<td>Free base</td>
<td>Free base</td>
<td>Sodium</td>
<td>Chloride</td>
</tr>
<tr>
<td>Structure</td>
<td>Macroporous styrene-divinylbenzene</td>
<td>Macroporous styrene-divinylbenzene</td>
<td>Macroporous styrene-divinylbenzene</td>
<td>Macroporous styrene-divinylbenzene</td>
<td>Macroporous styrene-divinylbenzene</td>
<td>Macroporous styrene-divinylbenzene</td>
</tr>
<tr>
<td>Physical form</td>
<td>Spheres</td>
<td>Uniform spheres</td>
<td>Spheres</td>
<td>Uniform spheres</td>
<td>Spheres</td>
<td>Spheres</td>
</tr>
<tr>
<td>U.S. standard mesh (typical)</td>
<td>16-40</td>
<td>-30 + 40 (95%)</td>
<td>16-50</td>
<td>-30 + 40 (95%)</td>
<td>16-35</td>
<td>16-50</td>
</tr>
<tr>
<td>Total capacity</td>
<td>1.8 meq/ml, min</td>
<td>1.8 meq/ml, min</td>
<td>1.7 meq/ml, min</td>
<td>1.60 meq/ml, min</td>
<td>1.8 meq/ml, min</td>
<td>1.2 meq/ml, min</td>
</tr>
<tr>
<td>Weak base capacity</td>
<td></td>
<td>1.50 meq/ml, min</td>
<td></td>
<td>1.35 meq/ml, min</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water retention capacity (typical)</td>
<td>42-48%</td>
<td>42-50%</td>
<td>40-50%</td>
<td>40-50%</td>
<td>42-48%</td>
<td>48-56%</td>
</tr>
<tr>
<td>Swell, %</td>
<td>~ 5% Na → H form</td>
<td>~ 5% Na → H form</td>
<td>~ 22% Free base → HCl</td>
<td>~ 20% Free base → HCl</td>
<td>~ 5% Na → H form</td>
<td>12% typical Cl⁻ → OH⁻</td>
</tr>
</tbody>
</table>
Pressure drop as a function of flow rate

The following charts are provided to help you determine pressure drop across beds of DOWEX resins. Possible causes for excessive pressure drop are discussed on Page 16.

**Figure 18 – Pressure drop with DOWEX 88 resin**

![Graph showing pressure drop with DOWEX 88 resin](image)

**Figure 19 – Pressure drop with DOWEX 66 resin**

![Graph showing pressure drop with DOWEX 66 resin](image)

**Figure 20 – Pressure drop with DOWEX MONOSPHERE 88 and MONOSPHERE 77 resins**

![Graph showing pressure drop with DOWEX MONOSPHERE 88 and MONOSPHERE 77 resins](image)
Figure 21 – Pressure drop with DOWEX 88 MB resin

Figure 22 – Pressure drop with DOWEX 22 resin
Backwash expansion characteristics

The backwash expansion curves in this section are provided to help you determine the expansion of your beds at a given temperature and flow rate. Colder water will expand the resins higher in the bed for a given pump rate.

Backwash expansion should be monitored carefully since insufficient expansion will decrease regeneration efficiency. Excessive expansion may lead to resins escaping the bed - a particular concern with anion resins, which are lighter than cation resins. More information on resin backwashing and recommendations for bed expansion are given on Page 4.
To determine flow rate at temperature \( t \) ° Fahrenheit:
\[
F^\circ_{\text{Fahrenheit}} = F_{77} \left[1 + 0.008 (t \text{ ° Fahrenheit} - 77)\right]
\]

To determine flow rate at temperature \( t \) ° Celsius:
\[
F^\circ_{\text{Celsius}} = F_{25} \left[1 + 0.014 (t \text{ ° Celsius} - 25)\right]
\]
To determine flow rate at temperature \( t \):

\[
F_{t}^\circ \text{Fahrenheit} = F_{77} \times (1 + 0.008(t - 77))
\]

\[
F_{t}^\circ \text{Celsius} = F_{25} \times (1 + 0.014(t - 25))
\]
Storage and Handling

Storage
For long shutdowns, cation and anion deashing resins can be stored in place in a manner that provides protection from microbial growth. The following recommendations will also increase the probability of a trouble free start-up.

Cation and anion deashing resin preparation and storage
1) Backwash the bed to a minimum of 50% expansion for as long as it takes to produce a clear and colorless effluent.
2) Clean up the resin by passing 2 bed volumes of 4% (1N) NaOH through the bed; rinse to neutral pH; pass through 1.5 bed volumes of 7% (2N) HCl; rinse to neutral pH.²
3) Pass 4% NaOH through the bed until at least a 0.5% (0.1N) concentration is detected in the effluent. The entire vessel should be full of 0.5% (minimum) NaOH solution for protection and cleaning of the dome space.
4) During the storage period, check the NaOH solution periodically by draining some off the bottom of the vessel. Replace the entire solution volume with fresh 0.5% NaOH if there is significant color development.

Bringing deashing units back on-line
1) Rinse off the NaOH storage solution to neutral pH.
2) Cation resins - Regenerate with a minimum of 1.5 bed volumes of 7% HCl; rinse to neutral pH. Anion resins - Cross-clean first with 7% HCl; rinse to neutral pH; regenerate with a minimum of 2.2 bed volumes of 4% NaOH; rinse to neutral.
3) Follow normal procedures from this point on.

Handling
WARNING: Oxidizing agents such as nitric acid attack organic resins under certain conditions and could result in a slightly degraded resin up to an explosive reaction. Before using strongly oxidizing agents, consult sources knowledgeable in handling such materials.

²Pumping rates of the chemicals should be such that there is a minimum contact time of 45 minutes.
How to get more information on DOWEX products and Dow support services

To learn more about DOWEX products, Dow technical support services, request additional literature, or to get help resolving a particular problem, simply call us toll-free at 1-800-447-4369 or contact your Dow technical service representative. You'll talk with someone who understands your needs and can provide the prompt, personal service you deserve.

Warning: Oxidizing agents such as nitric acid attack organic ion exchange resins under certain conditions. This could lead to anything from slight resin degradation to a violent exothermic reaction (explosion). Before using strong oxidizing agents, consult sources knowledgeable in handling such materials.

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