DuPont™ AmberLite™ CR99 Chromatography Resins for Starch Sweetener Purification

Uniformly Exceptional Performance
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This brochure was written for sweetener refiners and engineers. Inside you will find an overview of sweetener purification, with a focus on chromatographic resin selection, care and service cost reduction.
Elevate efficiencies, lower costs.

DuPont’s chromatographic separation resins are engineered to optimize performance in your plant, increasing productivity and reducing costs. DuPont is here to help you make the most of your resources, meet growing global sweetener demand, and achieve your operating goals.

DuPont™ AmberLite™ CR99 Resins

DuPont consistently offers:

- **Reliability** – capital investment in worldwide production facilities to supply increasing global demand and offer leading quality, global service, and support.
- **Value** – products designed for applications that help lower operating costs, increase throughput, yield, and product quality.
- **Innovation** – R&D focused on delivering innovative products to maximize plant performance.
Feeding a Growing Need

Producing more with less.

With a rising global middle class and an ever-increasing world population, there will be a need to make more food with fewer resources such as water and energy. This means your purification processes need to be more efficient than ever before! DuPont is committed to helping you reach this goal with world-class chromatographic separation resins – letting you achieve more with less.

By 2030, we will need:
- 30% more Water
- 45% more Energy
- 50% more Food

Source: World Business Council on Sustainable Development
An expanding sweetener market.

The global sweetener market is expected to grow at an average annual rate of 3%. Chromatographically-enriched sweeteners such as high fructose corn syrup, beet sugar (sucrose), and sugar alcohols were valued at $10 billion USD in 2014. While the high fructose corn syrup market growth is relatively flat, beet sugar and low-caloric sugar alcohols are expected to grow at 4% and 6%, respectively.

Chromatographically-Enriched Sweeteners with Applications

<table>
<thead>
<tr>
<th>Category</th>
<th>Sweetener</th>
<th>Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beet Sugar (Sucrose)</td>
<td>White Sugar</td>
<td>General Sweetener, Baking, Fermentation</td>
</tr>
<tr>
<td></td>
<td>Brown Sugar</td>
<td>Baking, Colorant, Flavoring Agent</td>
</tr>
<tr>
<td></td>
<td>Invert Syrup</td>
<td>Beverages, Jams, Ice Cream</td>
</tr>
<tr>
<td>Caloric Sweeteners</td>
<td>High Fructose Corn Syrup</td>
<td>Multi-Purpose Low-Cost Sugar Replacement, Beverages, Frozen Juices, Baked Goods, Tomato Products</td>
</tr>
<tr>
<td></td>
<td>Crystalline Fructose</td>
<td>Flavored Waters</td>
</tr>
<tr>
<td></td>
<td>Maltodextran</td>
<td>Food Filler, Thickener</td>
</tr>
<tr>
<td></td>
<td>Glucose Syrup</td>
<td>Hard Candies, Frozen Desserts, Gums, Jellies, Ice Cream</td>
</tr>
<tr>
<td></td>
<td>Dextrose</td>
<td>Food Powders, Toffees, Caramels</td>
</tr>
<tr>
<td>Sugar Alcohols</td>
<td>Sorbitol</td>
<td>Toothpaste, Sugar-Free Gums and Candies</td>
</tr>
<tr>
<td></td>
<td>Mannitol</td>
<td>Breath Fresheners, Diabetic Foods</td>
</tr>
<tr>
<td></td>
<td>Erythritol</td>
<td>Non-Caloric Sweetener, Typically Paired with High-Intensity Sweeteners</td>
</tr>
<tr>
<td></td>
<td>Maltitol</td>
<td>Sugar-Free Gums, Candies, Baked Goods, Ice Cream</td>
</tr>
</tbody>
</table>


Estimated Global Market for Chromatographically-Enriched Sweeteners

<table>
<thead>
<tr>
<th>Category</th>
<th>Market Share</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beet Sugar</td>
<td>18%</td>
</tr>
<tr>
<td>High Fructose Corn Syrup</td>
<td>21%</td>
</tr>
<tr>
<td>Sugar Alcohols</td>
<td>61%</td>
</tr>
</tbody>
</table>

Corn sweetener process.

A simplified corn sweetener refining process diagram is shown in Figure 1 on Page 7. Here, corn is milled down and digested with a combination of acids and/or enzymes to yield a mixture of dextrose and residual solids. This slurry is filtered, decolorized (organics removed), and then deashed to give a dextrose intermediate which can either be isomerized into fructose or chromatographically-enriched to produce crystalline dextrose.

Glucose isomerase converts 42-46% of the dextrose into fructose and a second deashing step is used to remove salts added as cofactors for the isomerase. A chromatographic separator, incorporating simulated moving bed technology, is used to enrich the fructose to 75-95%. Depending on the fructose purity, the enriched fructose is either crystallized or blended back with 42% high fructose corn syrup to yield 55% high fructose corn syrup suitable for use as a sweetener for soft drink bottlers. Additional polishing steps at the end of the refining process can enhance syrup quality through additional protein, color, and ash reduction.
Simplified Corn Sweetener Refining Process

Figure 1: Process flow diagram for a typical high fructose corn syrup plant.
Note: Dextrose is also known as glucose.
Process Technology

Chromatographic enrichment increases product purity.

For high fructose corn syrup (HFCS) production using current enzyme technology, the conversion of glucose (also known as dextrose) to fructose is economically limited to 42–46% fructose on a dry weight basis. Higher fructose purities, such as 55% high fructose corn syrup, are achieved through chromatographic separation. The process for making 55% HFCS is shown in Figure 2 below.

To make 55% HFCS, 42% syrup is separated chromatographically into a high-purity fructose stream and a high-purity glucose stream. Blending the high fructose stream with additional 42% syrup results in a 55% HFCS product suitable as a sweetener for soft drink bottlers. In other cases, the high fructose stream is used directly as a sweetener, e.g. the production of crystalline fructose. Using chromatographic separation, fructose can be purified to over 90%. The DuPont™ AmberLite™ CR99 Chromatographic Separation Resin product family is used in the calcium salt form for HFCS enrichment.

AmberLite™ CR99 Chromatographic Separation Resins in the potassium salt form are used in the production of high purity dextrose and beet sugar (sucrose). In these applications, a mixture of the target sugar (dextrose or sucrose), salts, and larger oligosaccharides are separated by size and ionic exclusion. The salts and oligosaccharides quickly exit the resin bed, and the slower-eluting dextrose or sucrose is enriched and removed of these contaminants.

SMB chromatography for continuous separation.

Figure 3 represents the simulated moving bed (SMB) operation commonly used in corn sweetener production for continuous chromatographic separation. The loop, represented as a single box above, consists of either a single column with distribution plates between sections or multiple columns piped in a loop. The loop is filled with resin beads and has multiple inlets and outlets for feed stream addition and product removal.

Feed (42% HFCS) and eluent (water) are continuously added at various inlets controlled by a computer control system. Simultaneously, extract (enriched fructose) and raffinate (primarily glucose) are withdrawn from the zones of high purity that result as the syrup moves through the column loop. The SMB process allows continuous separation of sugars with efficient resin utilization and low water consumption.

Figure 2: Chromatographic enrichment of high fructose corn syrup. Note: Stream compositions may vary from those shown due to plant-specific circumstances.
Chromatographic Separation Resin Technology

The right resin for any system.

DuPont has an array of resins appropriate for use in chromatographic separation systems, depending on system design, intended product mix, and purity requirements. The DuPont™ AmberLite™ CR99 Chromatography Resin product family has products ranging from 220 to 350 microns in particle size. Larger resin, such as AmberLite™ CR99/350 resin, is suited for deep-bed separation systems and is typically used in beet sugar processing.

AmberLite™ CR99/320 resin is a well-rounded “workhorse” resin offering good performance with low pressure drop. A typical application for this resin is high fructose corn syrup enrichment for 55% HFCS production. AmberLite™ CR99/310 resin offers higher performance, reduced water usage, and operational cost savings in many of the same applications as the 320-micron resin grade.

Now the AmberLite™ CR99 chromatography resin product line also includes 220- and 280-micron resins. AmberLite™ CR99/280 resin is specifically designed to maximize performance and minimize product dilution, while keeping pressure drop acceptable for many existing separation systems utilizing 310- or 320-micron beads. The extra performance minimizes water evaporation costs and is especially valuable in demanding sweetener separations such as high purity dextrose, crystalline fructose, specialty sugars, and polyol purification.

AmberLite™ CR99/220 resin is the smallest of DuPont’s chromatographic separation resins, delivering exceptional performance with minimal water usage. The small size of this product gives it faster interaction kinetics and more separating power over larger resins. AmberLite™ CR99/220 resin is designed for shallow-bed separation systems and systems capable of handling higher pressure drops from the smaller bead size.

Beet sugar manufacturing offers its own unique challenges and may require a larger resin size due to higher pressure drops caused by faster feed rates and deeper resin beds.

Figure 4: Comparative pressure drop data for different AmberLite™ CR99 Chromatography Resins in the calcium form. The pressure drops were measured in 50% dissolved solids (DS) high fructose corn syrup at 60°C.
Chromatographic Separation Resin Technology

Most DuPont™ AmberLite™ CR99 Chromatography Resins are available in two ionic forms – calcium and potassium. Calcium-form (Ca) chromatography resins are used in high fructose corn syrup purification, taking advantage of the differential affinity of fructose and dextrose for the calcium ion. Potassium-form (K) resins are used in size-exclusion chromatography to fractionate larger or smaller saccharides and remove residual salts. Typical applications of potassium-form chromatography resins include sucrose recovery from molasses in beet sugar and dextrose enrichment (Table 1).

The AmberLite™ CR Resin Advantage.

DuPont’s uniform particle size technology gives AmberLite™ CR99 Chromatography Resins exceptional uniformity in both size and quality. This uniformity helps sweeteners diffuse in and out of each bead at the same rate each time. The result is a sharper chromatographic wavefront with less broadening and dilution, which can keep separator water usage low and operational performance high.

DuPont’s uniform particle size technology also offers the advantage of reduced pressure drop. AmberLite™ CR99 resins lack the fine- and coarse-sized beads that reduce packed bed void volume, choke off flow through separators, and increase system pressure drop. Separators using AmberLite™ CR99 resins realize lower pressures than comparable beads of the same average size. This means that you can get lower system pressure at a given resin bead size, or better performance using a smaller sized resin, with the same system pressure.

<table>
<thead>
<tr>
<th>Chromatography Resin</th>
<th>Product Feature</th>
<th>Typical Applications</th>
<th>Separator Screen Size (µm)</th>
<th>Ionic Forms</th>
</tr>
</thead>
<tbody>
<tr>
<td>AmberLite™ CR99/350</td>
<td>Low Pressure Drop</td>
<td>Beet Sugar</td>
<td>175</td>
<td>K</td>
</tr>
<tr>
<td>AmberLite™ CR99/320</td>
<td>Standard “Workhorse” Product</td>
<td>HFCS, Beet Sugar, Dextrose</td>
<td>160</td>
<td>Ca, K, Na</td>
</tr>
<tr>
<td>AmberLite™ CR99/310</td>
<td>Reduced Operating Costs</td>
<td>HFCS, Sugar Alcohols, Beet Sugar</td>
<td>155</td>
<td>Ca, K, Na</td>
</tr>
<tr>
<td>AmberLite™ CR99/280</td>
<td>Difficult Separations, Reduced Separator Water Usage</td>
<td>HFCS, Sugar Alcohols, High Purity Fructose, Polyols</td>
<td>140</td>
<td>Ca, K</td>
</tr>
<tr>
<td>AmberLite™ CR99/220</td>
<td>Exceptional Performance in Shallow-Bed Separators, Operating Cost Reduction</td>
<td>High Purity Fructose, Sugar Alcohols, Polyols, Difficult to Separate and High Value Sweeteners</td>
<td>110</td>
<td>Ca</td>
</tr>
</tbody>
</table>

Table 1: DuPont sweetener resin products. Other ionic forms may be available - please contact DuPont to discuss your needs.

Figure 5: The AmberLite™ CR99 Chromatography Resins product line. Each image was taken at the same magnification.
Operational performance and cost savings.

The major cost in separator operation is the cost of evaporating water from product cuts to concentrate them to the desired percent dissolved solids (%DS). In most cases, resin and equipment costs are a small fraction of the evaporation and energy costs over the lifetime of the resin. The lowest total cost of resin ownership (cost of resin + operational costs) typically comes from using the highest performing resin that still gives acceptable pressure drop in the SMB separator.

DuPont™ AmberLite™ CR99 Chromatography Resins are optimized to provide excellent separations performance for a given SMB pressure specification, at lower operating costs. AmberLite™ CR99 Ca/220 resin, for example, can save 20% or more on separator water usage and energy costs vs. AmberLite™ CR99 Ca/320, provided the separation system can accommodate the higher pressure drop from using smaller beads.

Table 2 illustrates chromatographic separator performance and water usage at two different resin bead sizes. Both AmberLite™ CR99 Ca/320 and AmberLite™ CR99 Ca/220 resins were tested using a pilot four-column sequential simulated moving bed (SSMB) system for HFCS enrichment. In this test the syrup feed rate and target fructose purity were held constant to illustrate how smaller resin bead sizes improve process separations performance through lower eluent usage and higher fructose recovery.

Table 3 shows the operational cost savings that can come with using a smaller bead size. While smaller chromatographic resins generally have higher up-front costs, the operating savings realized over the resin’s lifetime offer to lower the total cost of owning such smaller resin grades. The key is to purchase the right resin for a given system, taking into account pressure limitations and the total cost of ownership. In this example, AmberLite™ CR99 Ca/220 resin can save over $250,000 per year in evaporation costs. Over a typical 5-10 year resin life, this adds up to well over $10 per liter of resin (or $280/ft³) of additional operating cost reduction on top of the established strong performance of AmberLite™ CR99 Ca/320.

<table>
<thead>
<tr>
<th>Resin</th>
<th>SMB Productivity</th>
<th>SMB Eluent Usage</th>
<th>Fructose Purity</th>
<th>Fructose Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>AmberLite™ CR99 Ca/320</td>
<td>98.0 kg dry product/m³ resin/h</td>
<td>106 kg water/kg dry product</td>
<td>88.8%</td>
<td>92.2%</td>
</tr>
<tr>
<td></td>
<td>61 lb dry product/ft³ resin/h</td>
<td>0.13 gal water/lb dry product</td>
<td>0.13 gal water/lb dry product</td>
<td>88.1%</td>
</tr>
<tr>
<td>AmberLite™ CR99 Ca/220</td>
<td>99.7 kg dry product/m³ resin/h</td>
<td>0.83 kg water/kg dry product</td>
<td>0.13 gal water/lb dry product</td>
<td>88.1%</td>
</tr>
<tr>
<td></td>
<td>6.2 lb dry product/ft³ resin/h</td>
<td>0.10 gal water/lb dry product</td>
<td>0.10 gal water/lb dry product</td>
<td>88.1%</td>
</tr>
</tbody>
</table>

Table 2: Sequential simulated moving bed (SSMB) performance of AmberLite™ CR99 99 Ca/320 and AmberLite™ CR99 Ca/220 Chromatography Resin in the separation of fructose and glucose using 60% DS 42% high fructose corn syrup as feed. Pilot separation tests were carried out using four 2.5-cm diameter columns. Actual system performance may vary, depending on scale and specific process conditions.

<table>
<thead>
<tr>
<th>Resin</th>
<th>Energy Cost ($/m³ Water Evaporated)</th>
<th>Evaporation Costs (cents/kg dry HFCS-55)</th>
<th>Annual Plant</th>
<th>Annual Savings ($/yr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AmberLite™ CR99 Ca/320</td>
<td>$5/m³</td>
<td>1.0</td>
<td>$1,028,500</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>$10/m³</td>
<td>2.0</td>
<td>$2,037,000</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>$15/m³</td>
<td>3.0</td>
<td>$3,055,500</td>
<td>–</td>
</tr>
<tr>
<td>AmberLite™ CR99 Ca/220</td>
<td>$5/m³</td>
<td>0.8</td>
<td>$805,500</td>
<td>$223,000</td>
</tr>
<tr>
<td></td>
<td>$10/m³</td>
<td>1.6</td>
<td>$1,595,000</td>
<td>$442,000</td>
</tr>
<tr>
<td></td>
<td>$15/m³</td>
<td>2.4</td>
<td>$2,392,500</td>
<td>$663,000</td>
</tr>
</tbody>
</table>

Table 3: Estimated process evaporation costs and savings for a HFCS plant using AmberLite™ CR99 Ca/320 and AmberLite™ CR99 Ca/220 Chromatography Resin. This calculation assumes separator performance from Table 2 above and that the plant operates 350 days/yr. Calculations are for a plant producing 100,000 MT/yr of dry HFCS-55. A plant of this size would use approximately 120 m³ of resin. Actual system performance may vary, depending on scale, specific process conditions, and cost inputs.
Chromatographic Separation Resin Technology

Separation mechanisms.

DuPont chromatography resins separate sweeteners using two different mechanisms – size/ion exclusion and ligand chromatography – depicted in Figure 6 below.

Size/Ion Exclusion: Potassium-form chromatography resins are used to separate sweeteners by size exclusion. The microporous resin structure allows smaller monosaccharides such as glucose (also known as dextrose) and fructose to diffuse into the resin, while blocking the entrance of larger oligosaccharides. This allows the beads to slow the progression of monosaccharides through the resin bed, while larger oligosaccharides travel through the packed resin bed more quickly. Salts are ionically excluded from the resin beads and also travel quickly through the resin bed.

Ligand Chromatography: Chromatographic separation resin beads in the calcium form create weak intermolecular complexes with sugars such as fructose and glucose that penetrate into the resin beads (Figure 6). The strength of this interaction is different for each sugar, depending on the chemistry (spatial orientation) of the hydroxyl groups in the sugar molecule. Fructose forms a stronger complex with calcium than glucose, causing the resin to slow down the progression of fructose more as the sugars move through the resin. Glucose moves faster through the resin bed and elutes sooner than fructose, resulting in separation of the two sugars.

Figure 6: Chromatographic separation of fructose from glucose (dextrose) and oligosaccharides. Oligosaccharides are excluded from the resin based on size and elute first, followed by glucose. Fructose elutes last due to stronger interactions with the calcium ion bound to the resin.
Chromatographic performance.

Chromatographic performance depends on a complex combination of variables including feed, equipment, operating parameters, and resin selection. In general, separation performance is a trade-off between purity, product recovery, system throughput, and operational costs such as eluent usage and evaporation. For example, higher product purity usually can be obtained at a cost of increased water usage or lower product recovery.

Chromatographic separation resins that promote increased diffusional kinetics with the feed (smaller resins, higher moisture content, etc.) tend to have higher separation performance. Diffusional kinetics increase with decreasing bead size and increasing contact area with the liquid feed, which helps sugars to enter the beads. Once the sugars are inside, higher resin moisture content increases diffusion through the bead itself, allowing sugars to move around and better interact with the resin faster.

Performance must be balanced against other process needs such as pressure drop and oxidative stability, as well as your production and product quality goals. For example, the Blake-Kozeny equation predicts that system pressure drop increases approximately two-fold when bead size is decreased 33%. Also, higher resin moisture content, while increasing performance, also correlates with reduced resistance to oxidation and increased bead compressibility (which causes system pressure issues). Process conditions, feed composition, and resin age can further affect actual performance in the field.

Figure 7 shows the relationship between pressure drop and diffusional kinetics as a function of resin bead size. Contact DuPont to discuss your specific needs and find the right DuPont™ AmberLite™ CR99 Chromatography Resin for your plant.

Pressure drop.

Pressure drop is a key process consideration when selecting a chromatographic resin for an SMB process. In general, pressure drop is inversely proportional to the square of the resin bead diameter; so smaller resins will have a higher pressure drop. Other factors such as bead compressibility, particle size uniformity, and bead packing in the resin bed can also influence pressure.

Existing deep-bed SMB systems or systems working with viscous feeds may require larger resin beads, such as a 320- or 350-µm bead size to stay within equipment pressure limits. Systems designed for higher pressure limits or with shallow bed depth can take advantage of the faster separation kinetics of smaller beads such as AmberLite™ CR99 99/310, AmberLite™ CR99/280, or AmberLite™ CR99/220 Chromatography Resins.
Chromatographic Separation Resin Technology

Resin lifetime.

As a resin ages, its moisture level increases, its ionic capacity decreases, and the beads swell and become softer. This is due to oxidative decrosslinking of the organic polymer chains inside the beads. Over time this leads to increased system pressure drop from beads expanding in size and becoming more compressible. Eventually the pressure drop limits system productivity and the resin must be replaced.

Resin breakdown is rapidly accelerated by dissolved oxygen in the separator system, as shown in Figure 8. DuPont recommends keeping SMB dissolved oxygen levels below 0.1 ppm (with a maximum of 0.5 ppm) to maintain resin performance and maximize potential resin life. Resin decomposition is also catalyzed by certain transition metals (especially iron) that can become ionically bound to the resin, so deashing upstream of the chromatographic separator is important.

DuPont System Optimization Services can help determine the condition of used resin and provide recommendations on maintaining or replacing resin.

Figure 8: Estimated resin lifetime for a typical chromatography resin as a function of system dissolved oxygen content. Actual resin lifetime can vary significantly from this estimate depending on specific process conditions.
Maintaining Chromatographic Separator Performance

Choosing a chromatography resin.

There are several properties to consider when selecting a chromatographic resin for a separation process. The primary parameters are:

**Bead Size**
The choice of bead size is a balance between separation performance and pressure drop. Smaller beads have faster separation kinetics but higher pressure drop. Most sweetener chromatography resins are 220–350 µm in diameter. Figure 7 shows the trade-off between separation kinetics and pressure drop as a function of bead size.

**Uniformity**
Uniform bead size is key to achieving high levels of separation performance with minimal pressure drop. Non-uniformity introduces tailing and peak broadening effects from non-uniform diffusional path lengths across larger and smaller beads. Non-uniform beads also have fines and coarse particles that can cause pressure and backwashing problems. A good chromatographic resin should have a uniformity coefficient less than 1.1. DuPont’s uniform particle size technology achieves these high uniformity standards.

**Water Retention Capacity**
This is a measure of how much water is present in the resin bead. Higher water retention capacities usually correlate with faster sugar diffusion and higher separations performance. However, higher water retention capacity also correlates with more compressible beads (causing higher pressure drop) and reduced resistance to oxidative damage. As resin ages, its water retention capacity will decrease.

**Total Exchange Capacity**
This indicates how much ion exchange functionality is in a given volume of resin. Total exchange capacity is inversely related to water retention capacity. As resin ages, its total exchange capacity will decrease.
Maintaining Chromatographic Separator Performance

Separator operation.

The major variable cost in separator operation is the cost of evaporating water from the product cuts to bring them to the desired percent dissolved solids (%DS). When a separator is new, it is recommended that the owner document the operating performance in terms of (1) capacity of product (such as 55% HFCS) produced per unit volume and (2) water used per pound dry product. These two numbers become the benchmark from which to compare the operating efficiency of the separator as it ages.

Separator tuning.

Small changes in recovery and purity can have a large impact on process economics and evaporation costs. The most common problem in uneconomical operation is poor separator tuning. Separator tuning is the delicate balance of feed and product flowrates, water usage, and separator valve switching to achieve a targeted separation.

SMB separators are complex. With the many variables that affect separator performance, most operators do not make changes unless there is a dramatic problem. SMB separators should be optimized by specially-trained engineers, as a small imbalance in operating parameters can cause large performance deficiencies. Careful and proper tuning can keep process performance at optimal levels and minimize production costs.

Poor tuning requires the plant to evaporate additional water to achieve the same product recovery, purity, and throughput. In more severe cases, product quality may be compromised. Regularly tuning up the separator as the equipment and resin ages is key to maintaining optimal performance. Proper resin selection for desired separator performance targets is also critical. Contact DuPont for help with optimizing your separator.

Maintaining your resin.

Chromatographic resins must be in the correct ionic form (e.g., calcium for fructose separation) for the resin to work properly. Poorly-deashed feeds can exchange sodium, magnesium, and iron into the resin. At low pH, hydrogen (H\(^+\)) ions can displace calcium from the resin. Hydrogen ions catalyze the formation of 5-hydroxymethyl-2-furfural (HMF) from fructose. Iron catalyzes oxidative degradation of the resin.

The resin should also be protected from premature aging (oxidation) by maintaining low dissolved oxygen levels in the separator. The dissolved oxygen level should be below 0.5 ppm, and ideally below 0.1 ppm.

The pH in the separator should be controlled to an optimum range of 4.0 – 6.5. Lower pH values will cause hydrogen to exchange onto the resin and will catalyze HMF formation. High pH values at high temperature can cause fructose degradation and by-product formation.

### Typical operating conditions for AmberLite™ CR99 Chromatography Resins

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Syrup temperature</td>
<td>60–71°C (140–160°F)</td>
</tr>
<tr>
<td>Recommended dissolved oxygen concentration</td>
<td>&lt; 0.1 ppm</td>
</tr>
<tr>
<td>Maximum dissolved oxygen concentration</td>
<td>0.5 ppm</td>
</tr>
<tr>
<td>Recommended pH</td>
<td>4.0–6.5</td>
</tr>
<tr>
<td>Simulated moving bed operation</td>
<td>with optimized tuning</td>
</tr>
</tbody>
</table>

*Table 4: Typical operating conditions for AmberLite™ CR99 Chromatography Resins.*
Maintaining Chromatographic Separator Performance

Typical operating conditions.

The DuPont™ AmberLite™ CR99 Chromatography Resin product family is designed with the operating conditions in Table 4 in mind. The crosslinked polymer structure of AmberLite™ CR99 Resins makes them resistant to acids, bases, most solvents, and temperatures up to 120°C.

Controlling HMF.

One of many UV-absorbing species found in corn syrups is 5-hydroxymethyl-2-furfural or HMF (also known as 5-hydroxymethyl-2-furancarboxaldehyde). It is thought to be a color precursor or a color marker, which indicates when a syrup could have high levels of heat color. HMF results from acidic dehydration of fructose. Its formation is promoted by high temperature and low pH.

The conditions for HMF formation are present in every chromatographic separator: high temperature, high fructose concentration, pH below neutral, and a long residence time in the separator. Maintaining effective process control using the conditions defined in Table 4 is key to minimizing HMF formation. Measuring baseline HMF levels in a separator system under normal process conditions can assist in troubleshooting when a process upset occurs.

Controlling microbiological growth.

In general, microbial growth occurs at cool to warm temperatures and dilute sweetener concentrations. Separators are normally maintained at 60°C (140°F) or above to inhibit microbial contamination. Elevated temperatures also keep syrup viscosities down and reduce system pressure drop.

The dissolved solids (DS) concentration inside the separator column varies with time and position in the column. High DS concentrations suppress microbial growth because very high sweetener concentrations are toxic to most microbes. However, there will be times and areas in the process of low DS where microbial growth could be very rapid if the temperature is not kept high. For short-term shutdowns, the high temperature will inhibit microbial growth. For shutdowns exceeding 12 hours, separators need to be sweetened-off thoroughly. Contact DuPont for advice on how to handle your resin in the event of a process upset.
DuPont Expertise

DuPont System Optimization Services™ (SOS)

Working with DuPont is easy and convenient. Whenever you choose DuPont resins, you get expert support from DuPont ion exchange technical service and development teams.

For more involved issues, DuPont offers a full range of System Optimization Services™ (SOS) to help you achieve optimal performance from your resin, system, and plant operations. SOS Services™ place our extensive knowledge and experience at your disposal. These services can complement your R&D innovation team, lighten the burden of your system start-up and staff training, and support the ongoing operation and maintenance of your system.

Request a Sample of DuPont Ion Exchange Products
Small orders of DuPont ion exchange resins, polymeric adsorbents, chelating resins, and copolymers can be ordered online through the Octochem website.
Reference and Contact Information

Other resin products for sweetener purification.

DuPont also manufactures a variety of resins for sweetener deashing, decolorizing and mixed bed polishing. The advantage of uniform particle size technology found in DuPont™ AmberLite™ CR99 Resins is available for many of these products to improve process kinetics and performance.

We are constantly innovating and developing new and improved resins. If you have an unmet process need, contact DuPont and let our scientists help you meet the demands of your process.

<table>
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<tr>
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<td>AmberLite™ FPA22</td>
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</table>

Table 5: DuPont ion exchange resin products used in the sweetener industry. Other ionic forms may be available - please contact DuPont to discuss your needs.

Resin properties and product data sheets.

The most current information on DuPont chromatographic separation resins and related products, including resin properties and product data sheets, is found on DuPont's website at:

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With a large global manufacturing footprint, strong R&D expertise and technical support services and systems, we supply high market volumes with high quality. DuPont partners with you, our customer, to understand unmet needs and develop tailored solutions.

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