APPLICATION GUIDE

Laboratory testing of ion exchange and adsorbent resins

This Application Guide presents information on how to carry out lab-scale process evaluations of ion exchange and adsorbent resins.
LABORATORY TESTING

This Application Guide presents information for conducting laboratory-scale tests on the effectiveness of ion exchange and adsorbent resins. For more detailed information on any product, go to www.purolite.com or contact the closest Purolite regional office to you listed on the back cover.

INTRODUCTION

Founded in 1981, Purolite is a leading manufacturer of ion exchange, catalyst, adsorbent and specialty resins. With global headquarters in the United States, Purolite is the only company that focuses 100% of its resources on the development and production of resin technology.

Responding to the needs of our customers, Purolite has built the largest technical sales force in the industry, the widest variety of products and five strategically located Research and Development groups. Our ISO 9001 certified manufacturing facilities in the U.S.A, Romania and China, combined with more than 40 sales offices in 30 countries, ensure complete worldwide coverage.

PREMIER PRODUCTS

The quality and consistency of our products is fundamental to our performance. Throughout all Purolite plants, production is carefully controlled to ensure that our products meet the most stringent criteria, regardless of where they are produced.

RELIABLE SERVICE

We are technical experts and problem solvers. Reliable and well trained, we understand the urgency required to keep businesses operating smoothly. Purolite employs the largest technical sales organization in the industry.

INNOVATIVE SOLUTIONS

Our continued investment in research & development means we are always perfecting and discovering innovative uses for ion exchange resins and adsorbents. We strive to make the impossible possible.
This guide describes how to perform a lab-scale process evaluation on the effectiveness of ion exchange (IEX) and adsorbent resins. Test results obtained and the depth of work performed may enable further operating condition optimization. While the test may not provide sufficient information to design a full-scale plant, it should show if a process is viable and enable the design of a larger pilot plant, if necessary.

Purolite is sensitive to the amount of time and expense dedicated to developing new and novel products and processes. If assistance is needed for resin configuration or system design optimization during the lab study, Purolite technical experts can be utilized, and confidentiality agreements can be arranged.

**Equipment**

To carry out initial trials, a 25 mm diameter glass column with a sintered glass bottom is ideal. However, a 25 mm diameter glass or plastic column is suitable to create a basic test system. If this is used, configure the column with a rubber stopper at each end, with a glass tube running through each, see Figure 1. A nylon cloth or screen should be placed over the surface of each stopper to retain the IEX or adsorbent resin in the bed. Then, fill the column to a depth of 15 – 25 mm with small diameter glass beads (3 mm diameter) to help distribute the fluid and hold the resin above the bottom of the column, preventing it from blocking the outlet.

If the column does not have a valve connection to control the flow rate at the exit end of the column, rubber tubing with a screw clamp assembly can be used as an on/off valve.

Depending on the laboratory set-up, additional equipment may include a funnel, tubing, a pump, nylon cloth or glass wool, and a graduated cylinder. In sensitive applications, the equipment should be sterilized and then fully rinsed with high-quality demineralized water before loading the resin.

**Figure 1 – Sample test column set-up for lab-scale process evaluations**
**Equipment set-up**

Depending on the nature of the trial, the column can be fed either by gravity or by a peristaltic or diaphragm pump. If gravity feed is used, arrange the pipework from the column in a “U” shape so that it rises to a level above the top of the bed, keeping the flooded at all times.

Whether gravity or pumped systems are used, it is essential to have solutions of regenerant and demineralized water pre-prepared and ready for use before the trial and regeneration processes begin.

**Storage**

If IEX or adsorbent resin is stored prior to testing, make sure the containers are not left open to the atmosphere or allowed to dry out. Keep storage containers away from strong sunlight and hot or cold temperatures.

**Non-aqueous applications**

If testing the sample in a non-aqueous application, the IEX or adsorbent resin should be used in the dried form for best results. This will prevent water contamination of the treated product. Resin samples received in the hydrated form should be pre-conditioned to remove the water by displacing with suitable solvents such as acetone or alcohol, if permitted in the specific process.

**Rinsing**

When samples are sent out from Purolite, the resin is either taken from production batches or from warehouse stock. Resin that is stored for any length of time will require rinsing with demineralized water to reduce leachables before trials can be performed. In many applications, 5 – 10 bed volumes should be sufficient.

High-purity applications may require different preparation. Please contact your regional Purolite sales office for assistance.

**Preconditioning**

Prior to running a laboratory test, the sample needs to be conditioned to ensure full swelling and hydration of the polymer. Unless otherwise advised, the polymer must be soaked.

Do not load IEX or adsorbent resin in a dry column. Fill \( \frac{1}{2} \) to \( \frac{1}{3} \) of the column in advance with deionized water, then transfer the pretreated IEX or adsorbent resin into the column.

A minimum total bed depth of 760 mm should be maintained for the trial. In most applications, the process will improve if the bed depth is increased.

**Resin volume**

Approximately 125 ml of resin is sufficient for an initial test of a single resin bed. If the inlet load is very low, it will require a large amount of solution to be processed through the bed to reach exhaustion, and greatly increase the time it takes to complete each test.

**Backwashing**

Once the column is loaded, the resin should be backwashed with demineralized water in an up-flow direction for 10 – 15 minutes to fully classify the bed. During settlement, the resin bed will expand and the larger particles will fall towards the bottom of the bed while the smaller beads will locate nearer the surface.

Allow the IEX or adsorbent resin to settle for approximately 5 minutes.

Following this process, and depending on the particle size range of the resin, the height of the resin will increase. This resin height or bed volume (BV) must be noted and should be used for all test calculations going forward.

Next, drain the column to leave a maximum of 1 cm of water remaining above the bed and discard the initial run-off. Then, begin feeding the solution into the column. Adjust the opening of the bottom valve on your column set-up to control the total flow through the resin.
Sampling

It is important to routinely sample the resin during service runs to enable adjustments to pH. Levels of pH can drop significantly in the treated water, affecting alkalinity, breakthrough behavior, leakage and run lengths. Frequent analysis for the target analyte will also reveal critical information about column loading.

Process parameters

Depending on the application, the process conditions for the resin will vary widely. For processes that are not fully developed, discovering and maintaining the optimum specific flow rate will be one of the primary objectives of the laboratory study.

In many water applications where the loading is small and conventional ion exchange is taking place, flow rates through the resin can be high (Up to 50 BV/h)—and sometimes even greater flow rates are used. In special process applications, or where highly selective removal is required, flow rates can be much lower (1 – 10 BV/h).

Resin regeneration is normally carried out at relatively low flow rates (1 – 6 BV/h) to achieve maximum removal from the beads, and is followed by a slow rinse at a similar flow rate to maximize removal of the regenerant.

The final rinse is carried out using a higher flow rate than the in-service flow rate.

In non-aqueous process applications, the resin is often regenerated in the aqueous state. Under these circumstances the process liquor must first be displaced with water. This is often referred to as “sweetening off,” and after regeneration the water is displaced with process liquor and is called “sweetening on.” These terms derive from the sugar industry where IEX and adsorbent resin is widely used.

Service operation

Once a trial run begins, the resin should be allowed to continue to operate through to exhaustion. The experiment should not be stopped mid cycle. Most ion exchange reactions are reversible, and once the solution is stopped it tries to reach equilibrium. When this happens, the ions come back off the resin into solution. This can cause premature resin exhaustion and false results.

Under normal condition tests, the bed must remain covered with solution. Never drain the column and introduce air into the bed. Air bubbles are difficult to remove and will result in poor test performance.

Three consecutive cycles producing consistent results should be obtained before changing any operating conditions to optimize performance.

It normally takes two or three cycles to obtain reliable test information.

Table 1 – Suggested parameters for testing viability of ion exchange and adsorbent resin

<table>
<thead>
<tr>
<th>PARAMETER</th>
<th>MINIMUM MEASUREMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin volume</td>
<td>≥ 125 ml (125 ml is most typical)</td>
</tr>
<tr>
<td>Resin bed depth</td>
<td>250 – 600 mm (classified, minimum)</td>
</tr>
<tr>
<td>Service flow rate</td>
<td>2 – 50 BV / h (8 – 20 BV / h typical)</td>
</tr>
<tr>
<td>Regenerant flow rate</td>
<td>2 – 6 BV / h (2 BV / h typical)</td>
</tr>
<tr>
<td>Regenerant contact time</td>
<td>15 – 60 minutes (≥ 30 minutes preferred)</td>
</tr>
<tr>
<td>Slow displacement rinse</td>
<td>1 – 2 BV</td>
</tr>
<tr>
<td>Final fast rinse</td>
<td>2 – 10 BV</td>
</tr>
</tbody>
</table>

For regeneration chemicals, concentrations, quantities and details for sweetening on and off, contact your local Purolite technical sales professional for guidance.
Regeneration

Many different ion exchange regeneration processes have been developed for aqueous and non-aqueous applications.

The simplest, most common method of regeneration is co-flow (often referred to as co-current), where the regenerant passes down through the resin in the same direction as the service flow.

In co-flow regeneration, the first stage of every regeneration cycle is to backwash the bed to relieve compaction and remove suspended matter. Solutions or water containing more than trace levels of solids should be removed by filtration prior to the test column. Regeneration followed by a slow displacement rinse and then final rinse can then be carried out.

A more efficient and thorough regeneration can be achieved using a technique called counter-flow regeneration (often referred to as counter current or reverse flow). Here the regenerant passes in the opposite direction to the service flow, resulting in much lower leakage from the bed. Counter-flow regeneration is somewhat difficult to set up in the laboratory and parameters such as bed depth are more critical.

Additionally, it is important to prevent the bed from fluidizing when regenerant is passed in an upflow direction. To prevent fluidization, nylon cloth or glass wool can be placed on top of the resin to fill the freeboard space. This will stabilize the column and prevent beads from rising. Guidance from a Purolite technical expert is recommended.

In laboratory work, demineralized water is preferred for regenerant dilution and the rinse stages. This will not only make capacity calculations easier, but also ensure there is no ionic loading of the resin.

Benefits of lab-scale testing

Controlled lab-scale column trials generate useful, meaningful data on resin effectiveness for a given process, and can become the basis of a successful full-scale system. Evaluating all steps within a controlled environment—including bed preparation, loading, feed displacement, rinse and regeneration—is the most reliable and economical way to determine if resin will perform as expected. Lab trials also provide an opportunity to proactively make adjustments to regenerant concentration, flow rate and other parameters affecting resin performance prior to committing to a full-scale operation.