Laboratory Testing of Ion Exchange and Adsorbent Resins

This Best Practices Guide presents information on how to carry out lab-scale process evaluations of ion exchange and adsorbent resins.
About Purolite

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

Responding to our customers’ needs, Purolite has the widest variety of products and the industry’s largest technical sales force. Globally, we have strategically located research and development centers and application laboratories. Our ISO 9001 certified manufacturing facilities in the USA, United Kingdom, Romania and China combined with more than 40 sales offices in 30 countries ensure complete worldwide coverage.

Purolite has been part of Ecolab since 2021. A trusted partner at nearly three million commercial customer locations, Ecolab (ECL) is the global leader in water, hygiene and infection prevention solutions and services. Ecolab delivers comprehensive solutions, data-driven insights and personalized service to advance food safety, maintain clean and safe environments, optimize water and energy use, and improve operational efficiencies and sustainability for customers in the food, healthcare, hospitality and industrial markets in more than 170 countries around the world.

PREMIER PRODUCTS

The quality and consistency of our products are 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 team in the industry.

INNOVATIVE SOLUTIONS

Our continued investment in research and development means we are always perfecting and discovering innovative uses for ion exchange resins and adsorbents. We strive to make the impossible possible.
Laboratory Testing of Ion Exchange and Adsorbent Resins

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Introduction to Laboratory Testing of Ion Exchange Resins

At Purolite, we are sensitive to the amount of time and expense dedicated to developing new products and processes. We offer this guide which describes how to perform a lab-scale process evaluation on the effectiveness of ion exchange resins. Test results obtained and the in-depth work performed may enable further operating condition optimization. Testing of a resin will provide the user with insight into how to improve that resin's effectiveness.

If assistance is needed for resin configuration or system design optimization during the lab study, customers can contact Purolite technical experts, and, if necessary, we can arrange confidentiality agreements.

We stick to a standard testing process for this guide, but keep in mind that alternative methods may be preferable depending on the equipment available or other similar factors. Controlled lab-scale column trials generate helpful, meaningful data on resin effectiveness for a given process and can become the basis of a successful full-scale system.

How to Test a Resin Video

![QR code]

Equipment Set-Up

While a 25 mm diameter glass column with a sintered glass bottom is ideal for conducting initial trials, a plastic column is suitable to create a basic test system. The recommended height of the column is 1200 mm. This will allow enough headspace for expansion if necessary. Configure the column with a rubber stopper at each end, with a glass tube running through the system (see Figure 1). Nylon cloth or screen should be placed over the surface of each stopper to retain the resin in the bed. Fill the column to a depth of 15–25 mm with small diameter glass beads (3 mm diameter) or sand 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, cotton or glass wool and a graduated cylinder. Depending on the trial, the column can be fed either by gravity or 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 resin flooded at all times.

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

FIGURE 1

Column Flow

1. 25 mm diameter x 1200 mm tall glass column with a sintered glass bottom
2. Rubber stopper
3. Glass tube
4. Nylon cloth or screen
5. Small diameter glass beads (3 mm diameter) or sand
6. Rubber tubing with a screw clamp assembly
7. Funnel
8. Tubing
9. Pump
10. Cotton or glass wool
11. Graduated cylinder
Sample Testing 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.

Flow Rates

Typical flow rates are 8–40 bed volumes per hour (BV/h) depending on the ionic concentration of the solution. In many aqueous applications where the impurity concentration is small and conventional ion exchange occurs, flow rates through the resin may be as high as 50 BV/h. In special process applications, or where highly selective removal is required, flow rates usually are much lower, between 1–10 BV/h.

Regeneration

Resin regeneration is typically carried out at relatively low flow rates of 1–6 BV/h to achieve maximum removal from the beads. A slow rinse follows this process at a similar flow rate to maximize the removal of the regenerant. The final rinse is carried out using a similar flow rate to the in-service flow rate.

Preparing the Resin Sample

Storage and Preconditioning

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

When samples are sent out from Purolite, the resin is taken directly from production batches or warehouse stock. If the resin is stored before testing, make sure the containers are not left open to the atmosphere or allowed to dry out. Keep resin storage containers away from strong sunlight and hot or cold temperatures.
Ion exchange resin 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.

### Resin Volume

Approximately 370–380 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.

### Loading

Fill 1/2 to 1/3 of the column in advance with deionized water, then transfer the pretreated ion exchange resin into the column. Maintain a minimum total bed depth of 760 mm (372 ml in described apparatus) for the trial. In most applications, the process will improve if the bed depth is increased. Do not load ion exchange resin into a dry column. Do not use the above procedure for mixed beds.

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<th><strong>TABLE 1</strong> Suggested Parameters for Testing Viability of Ion Exchange Resins</th>
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<tr>
<td><strong>Parameter</strong></td>
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For regeneration chemicals, concentrations and quantities, contact your local Purolite technical sales professional for guidance.
Running the Resin Sample Trial

Preparing the Column for Testing

Once the column is loaded, the resin should be backwashed with demineralized water in an up-flow direction for 10–15 minutes to classify the bed fully.

During backwash, the resin bed will expand, and the larger particles will fall towards the bottom of the bed while the smaller beads will be located near the surface.

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 used for all test calculations in the future.

Drain the column to leave a maximum of 1 cm of water above the bed. Discard the initial run-off.

Service Operation

Once a trial run begins, the resin should continue to operate to exhaustion or desired endpoint. The experiment should not be stopped mid-cycle, as most ion exchange reactions are reversible, and if the solution is stopped, it tries to reach an equilibrium. When this happens, the ions come back off the resin into solution. This can cause premature resin exhaustion and false results.

In tests with normal conditions, 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.

Sampling

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.

It is important to routinely sample the resin effluent during service runs to monitor performance. Frequent analysis of the target analyte will also reveal critical information about column loading. Additional testing may be for alkalinity, leakage, pH or other attributes as desired.
Regeneration of Resin Bed

The simplest, most common regeneration method 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) above trace solids levels should be filtered before entering the test column. The regeneration solution is passed through the resin at a slow rate to allow adequate contact time. This is followed by a slow displacement rinse and then a final fast rinse. In laboratory work, demineralized water is preferred for regenerant dilution and the rinsing stages. This will make capacity calculations easier and ensure there is no ionic loading of the resin.

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

Additionally, it is vital to prevent the bed from fluidizing when the regenerant is passed in an up-flow direction. Place cotton or glass wool on top of the resin to fill the freeboard space to prevent fluidization. This will stabilize the column and prevent beads from rising. Guidance from a Purolite technical expert is recommended.

Suggested Regeneration Parameters

- **Regenerant Flow Rate**: 2–6 BV/h (2 BV/h typical)
- **Regenerant Contact Time**: 15–60 minutes (> 30 minutes preferred)
- **Slow Rinse**: Normally same flow rate and time as the regenerant.
- **Fast Rinse**: Same flow rate as the service flow, typically 30 minutes.
Purolite, the leading manufacturer of quality ion exchange, catalyst, adsorbent and specialty high-performance resins, is the only company that focuses 100% of its resources on the development and production of resin technology.

We’re ready to solve your process challenges. For further information on Purolite products and services, visit www.purolite.com or contact your nearest Technical Sales Office.

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