

ENGINEERING BULLETIN

Purolite® C100 & C100H

Hydrogen Cycle Operation
Sulfuric Acid Regeneration

Purolite C100 & C100H are premium, industrial-grade gel polystyrenic strong acid cation exchange resins supplied in the Hydrogen form. The principal application in the Hydrogen form is water demineralization.



Purolite®

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PUROLITE® C100 and C100H HYDROGEN CYCLE SULFURIC ACID REGENERATION

Purolite C100 and Purolite C100H are industrial grade, premium gel, polystyrenic, strong acid cation exchange resins supplied in sodium and hydrogen form respectively. Their principal application is in water demineralization. Purolite C100, being supplied in the sodium form, needs to be converted to the hydrogen form by applying a double or triple acid regeneration. As the resin swells between the sodium and hydrogen form, this must be taken into account in any design calculations.

This document provides information and engineering data on the removal of cations as part of the demineralization process. On exhaustion, the resin can be regenerated with different acids. Those commonly employed are sulfuric acid and hydrochloric acid. This bulletin covers sulfuric acid regeneration. A separate bulletin is available on the performance using hydrochloric acid. For more details please consult the Hydrochloric Acid Regeneration Engineering Bulletin.

TYPICAL PHYSICAL AND CHEMICAL CHARACTERISTICS	
Polymer structure	Polystyrene crosslinked with DVB
Physical form	Amber, clear spherical beads
Functional groups	Sulfonic
Ionic form, as shipped:	Purolite C100 Purolite C100H
Total capacity, Na ⁺ form	2.0 eq/l (43.7 Kgr/ft ³) min.
Moisture retention, Na ⁺ form	44 - 48%
Particle size range	300 - 1200 µm 1% max. <300 µm
Uniformity coefficient	1.7 max.
Reversible swelling, Na ⁺ → H ⁺	8%
Specific gravity, Na ⁺ form	Approx. 1.29
Specific gravity, H ⁺ form	Approx. 1.22
Shipping weight, Na ⁺ form	800 - 840 g/l (50.0 - 52.5 lb/ft ³)

Available Grades

Purolite C100 and Purolite C100H are available in different grades. The list below indicates products supplied in the Na⁺ form, but H⁺ form versions are also available in most cases.

Single Bed Applications

- **Purolite C100** is a standard grade resin with a Gaussian particle size distribution in the range 300-1200 µm. Its principal application is in co-flow and traditional counter-flow regenerated plants, where classification of the bed inside the operating vessel is possible.
- **Purolite C100C** is a modified grade with a particle size in the range 400-1200 µm, for use in high flow rate applications where the standard grade resin would present an unacceptably high pressure drop across the bed.
- **Purofine® PFC100** is a Uniform Particle Size product with a mean particle size of 570 µm and a UC of 1.1-1.2, offering improved performance in softening and demineralization systems, with regard to capacity, leakage, pressure drop and rinse water requirements.

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- **Puorpack® PPC100** is another Uniform Particle Size product, offering similar advantages, but with a mean particle size of 650 µm. This product has been specifically developed for the PUROPACK® system and other packed bed counter-flow designs employing either up-flow or down-flow service operation. This resin has also been widely used in co-flow and other counter-flow engineering designs, including air hold down, split flow, water hold down, etc.. Both Puorfine PFC100 and Puorpack PPC100 have also seen successful operation in short cycle plants.
- **Purolite C100S** is a specially cleaned and trimmed food grade resin with a particle size in the range 400-1200 µm, for use in food processing, such as in the sugar industry.

Dual Layer (Stratified Bed) Applications

- **Purolite C100DL** is a specially designed, coarse grade resin, with a particle size range of 630-1200 µm. Its principal application is in layered bed cation exchange units in conjunction with a DL grade Purolite weak acid cation resin such as Purolite C104DLPlus.

Mixed Bed Applications

Three grades of **Purolite C100** are widely used in mixed beds. These are all specifically designed to separate well from anion components, whether the anion resins are gel or macroporous. These cation resins are usually delivered in H⁺ form.

- **Purolite C100MBH** is a modified, simple mixed bed grade resin with a Gaussian particle size distribution in the range 425-1200 µm. It is used with either gel or macroporous mixed bed grade anion resins such as Purolite A400MB, Purolite A600MB, Purolite A200MB, Purolite A500MB or Purolite A510MB.
- **Purolite C100TLH** is a specially graded resin with a relatively coarse particle size for use in TRILITE™ mixed bed systems, with or without intermediate inert, employing internal or external regeneration, in conjunction with a suitable TL grade anion resin, such as Purolite A400TL or Purolite A500TL.
- **Puorpack® PPC100H** is a Uniform Particle Size product, normally used in mixed bed applications with a slightly finer, uniform particle size Puorfine anion resin, such as Puorfine® PFA400MB or Puorfine® PFA500MB.

Typical Operating Information

Service Operation

In service operation water is normally pumped through the resin bed, which is retained within a pressure vessel. The vessel has top and bottom distribution / collection systems. These systems are designed to ensure the water passes evenly through the ion exchange bed in service operation. As the water passes through the resin, the cations (principally calcium, magnesium, sodium, potassium, iron and any other dissolved cations present) are exchanged with hydrogen ions. The decationized water has a higher hydrogen (H⁺) content, a lower pH and a higher conductivity. When the resin is exhausted it is then regenerated with an acid solution to put the resin back into the hydrogen form, ready for the next service operation. It is important for the internal systems within the cation unit to efficiently distribute and collect the water in service and the regenerant acid solution, rinses, etc., especially since the regenerant and rinse flow rates are usually much lower than the service flow rate.

In service operation optimum performance is achieved at service flow rates between 8 and 40 BV/h (Bed Volumes per hour) or 1 to 5 gpm/ft³ (US gallons per minute per cubic foot of resin) within linear flow rates (velocities) of 10 to 50 m³/m²/h (m/h) or 4 to 20 gpm/ft² (US gallons per minute per square foot of vessel cross-section), whereas acid regeneration is carried out at flow rates of 4 to 16 BV/h or 0.5 to 2.0 gpm/ft³. Within these limits internal distribution / collection systems can operate efficiently at the higher service and lower regenerant flow rates. At very low service flow rates channelling can occur within the resin bed resulting in poor plant performance and short capacity between regenerations. This is particularly likely when long service cycles are also employed.

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The ratio of height to diameter is important in any ion exchange unit design. While some small industrial demineralization plants operate with very shallow bed depths, bed depths below 610 mm (2 ft.) should be avoided and preferably bed depths greater than 1000 mm (3 ft. 3 in) employed. Vessel height and pressure drop are normally the controlling factor on the maximum height of the bed. For Purolite C100 we recommend that pressure drop across the bed should be maintained at less than 150 kPa (22 psi), having made allowance for bed compaction and any solids loading across a classified bed. Bed depths greater than 2500 mm (8 ft.) are rarely encountered.

Although smaller freeboards are commonly encountered, we recommend a minimum 75% freeboard (space) above the resin bed to allow at least 50% bed expansion during backwash. This is normally adequate for a co-flow regenerated vessel, and assures a good hydraulic classification of the resin bed. Fully classified beds have a higher void fraction which leads to lower pressure drop. This is particularly advantageous when high specific velocities are encountered.

Service operation is usually terminated by detection of increased conductivity at the exit of the anion column, due to increased sodium leakage from the cation bed. Occasionally, on large counter-flow regenerated plants, sodium monitors are employed on the outlet of the cation unit to initiate regeneration. The subsequent regeneration can be manually or automatically initiated via the control system.

While co-flow and traditional counter-flow regenerated plant designs allow backwashing of the resin bed within the service operation unit, they will only tolerate a low level of suspended solids present in the incoming water supply. The resins are not expected to work as a mechanical filter, and an adequate pre-treatment should always be included in the plant layout if optimum performance is to be achieved.

Regeneration

The resin regeneration can be performed either co-flow or counter-flow. The regeneration is termed co-flow when the regenerant flows through the resin bed in the same direction, normally downwards or “top to bottom”, in which the water flows during the service operation. When the regenerant flow is in the opposite direction to service flow, then the term used is counter-flow regeneration. Other terms such as co-current and counter-current are also used to describe these two principal regeneration techniques.

When counter-flow regeneration is employed, it is important to note that in the up flow stages (except backwash) the bed must remain static. Packed beds, air hold down, split flow and water hold down are just some of the systems employed to achieve this requirement.

In some counter-flow regenerated systems the design allows service flow to be upward through the bed and regeneration downwards. In such cases it is important that the bed must remain static throughout the service operation.

Co-Flow Regeneration

The co-flow regeneration technique is normally made up of 5 steps and typically takes between 1 and 2 hours depending on the detailed design. For this type of regeneration, the influent water is typically of adequate quality for all steps, including regenerant dilution.

The first step of co-flow regeneration is backwash. The backwash water enters the unit through the bottom collection / distribution system, loosening the bed and causing the bed to expand as the water passes up through it. The flow rate should be set for the freeboard available in the unit at the minimum water temperature. The backwash is designed to both decompact the resin, for better regenerant contact, as well as for removing any suspended solids that have been filtered out of the incoming supply and accumulated within the bed. The backwash water volume required will depend on the extent of solids loading. Where the bed only requires loosening for better regenerant contact then 1 FBV (free board volume) is normally sufficient. However, when filtered solids are present the volume required can be considerably greater. After the backwash a “bed settle” step is required.

The bed settle allows the resin to settle back and reform the static bed prior to regenerant injection. Depending on the size of the bed, free board, and backwash rate used, this step can take between 3 and 8 minutes.

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Regenerant injection at the correct flow rate and acid concentration are critical. Good contact between the acid solution and the resin is essential for optimum performance. However, for sulfuric acid regeneration of cation resins there are specific problems that can be encountered, which require special care to be taken. This is when calcium present in the influent water is exchanged by the resin and is then removed from it during regeneration. As all naturally occurring raw waters contain some calcium and some levels are quite high, particularly in ground waters, it is important that the following recommendations are complied with on all regenerations.

The use of sulfuric acid can easily result in the precipitation of calcium sulfate within the resin bed when the saturation point for calcium sulfate is reached. The more calcium present in the raw water, and present on the resin after service operation, the greater the risk of reaching the saturation point as the acid passes through the bed and for the precipitation to occur. Purolite, like other resin manufacturers, set down operating guidelines for sulfuric acid injection based on the calcium content of the incoming water supply. These are indicated in Figure 1.

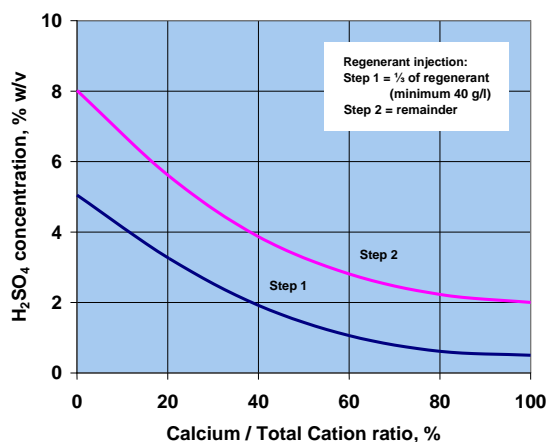
The lower the sulfuric acid concentration and the shorter the contact time, the lower the risk of precipitation occurring. However, to try to ensure that waste water volumes are not too great and to improve regeneration efficiency, the recommendations start with a low acid concentration which can be increased in stages during the injection period. This is referred to as step-wise regeneration.

Temperature also plays a part in precipitation. In hot climates increased temperature can increase the risk of precipitation and requires lower concentrations and shorter contact times.

For hydrochloric acid there is no such risk. The chloride salts of commonly encountered cations are far more soluble. Higher hydrochloric acid concentrations can be used and produce less effluent waste on regeneration. (See Purolite C100 & C100H - Hydrochloric Acid Regeneration Engineering Bulletin).

Experience shows that precipitation can occur quite easily. If the above guidelines are not followed, minor precipitation problems can sometimes be treated with a hydrochloric acid soak to re-dissolve the calcium sulfate and recover the resin. If the precipitation is severe, complete replacement of the resin is required because chemical clean-up is not a cost effective solution.

Figure 1 - Recommended maximum acid concentrations based on incoming calcium levels - Co-flow regenerated columns



The sulfuric acid regeneration level (amount of acid per litre or cubic foot of resin) will typically be between 80 and 160 g/l (5 – 10 lb/ft³), although regeneration levels as low as 60 g/l (4 lb/ft³) and as high as 240 g/l (15 lb/ft³) are sometimes employed. Please note all regeneration levels are expressed for the pure chemical (100%) strength. In order to calculate the exact volume of regenerant required per regeneration you need to know the acid concentration available on site.

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Sulfuric acid should be introduced step-wise at flow rates of 4 - 16 BV/h (0.5 - 2.0 gpm/ft³) and concentrations from 0.5 to 8.0% as indicated in Figure 1. The contact time between the resin and the regenerant solution should be minimum 30 minutes but not exceeding 1 hour.

The slow (regenerant displacement) rinse is always carried out at flow rates similar to the acid injection step. This is to ensure a uniform contact time between the resin and the regenerant solution and that the rinse water follows the same route of the regenerant through the resin bed. Since slow rinses are usually more efficient in removing the spent regenerant from the resin, using a longer slow rinse can reduce the amount of final rinse required at the end of the regeneration. Normally 1 - 3 BV (7.5 - 22.5 gal/ft³) of slow rinse are applied.

The final rinse is often carried out at the service flow rate. This also acts as a proving condition prior to returning to service after regeneration. On some occasions, where flow restrictions occur, the plant final rinse is carried out at a rate lower than the service flow rate. Normally 3 - 6 BV (22.5 - 45 gal/ft³) are required depending on the design of the distribution / collection systems and the amount of slow rinsing previously performed.

Table 1 - Typical Sulfuric Acid regeneration conditions for co-flow regenerated columns

Step	Design Basis	Duration
Backwash	Set for minimum water temperature to give 50% bed expansion. Refer to Figure 21 for details.	1 FBV on clean water supplies and 2-3 FBV where solids are present
Bed settle	To allow the bed to reform fully classified	3 to 8 minutes
H ₂ SO ₄ injection	Refer to Figure 1 showing recommended acid concentration dependent on calcium level in the raw water.	Typically 30-60 minutes depending on regeneration level and flow rate
Slow rinse	1-3 BV (7.5 to 22.5 gal/ft ³) at approx. regenerant flow rate	Typically 20-40 minutes depending on volume of water applied and flow rate
Final rinse	3-6 BV (22.5 to 45 gal/ft ³) preferably at service flow rate or alternatively > 15 BV/h (2 US gpm/ft ³)	Typically 10-20 minutes

(Key: BV = Bed Volume, BV/h = Bed Volume per hour, FBV = Free board volume above resin bed)

Counter-Flow Regeneration

Traditional counter-flow regeneration techniques normally have fewer steps than those described earlier for co-flow regeneration and typically take between 1 and 1½ hours depending on the detailed design. This type of regeneration requires, for some steps, the use of cation free water. Decationized or demineralized water must be used for the acid dilution / injection and slow rinse steps, if the published leakage is to be obtained. The water is either set aside during the previous service run or, in case of multi stream plants, it can be supplied by one of the other on-line streams. When decationized water is stored for regeneration, some plants use a dedicated tank, but when a degassing tower is part of the process, the degassing tower sump is normally used for this duty. When demineralized water is used, then the client's treated water tank or a separate tank are employed.

In a counter-flow regenerated system, the backwash step, which is always the first step of a co-flow regeneration, is not normally performed each cycle, but a means of carrying out periodic full bed backwashes, either inside the service unit or in external dedicated vessels, should always be included in the plant design. Some engineering designs allow for sub-surface backwashes to be carried out each cycle, but such partial backwashes should not be intended as a replacement of periodic full bed backwashes. After a full bed backwash the resin should always be regenerated with double the normal amount of acid to restore full counter-flow performance.

Similar to co-flow regeneration, special care needs to be taken with counter-flow regeneration to prevent calcium sulfate precipitation with regard to acid concentration and injection flow rate. In counter-flow regeneration the acid enters the bed from the opposite direction to the raw water and therefore encounters the sodium and potassium loaded resin before reaching the magnesium and calcium loaded areas. For these and other reasons, counter-flow regeneration conditions must be very much tailored to the plant design and very often engineering companies have

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their own “sets of conditions” which have been proved against that design. Purolite’s recommendations on acid concentrations and flow rates are therefore provided as general guidance only.

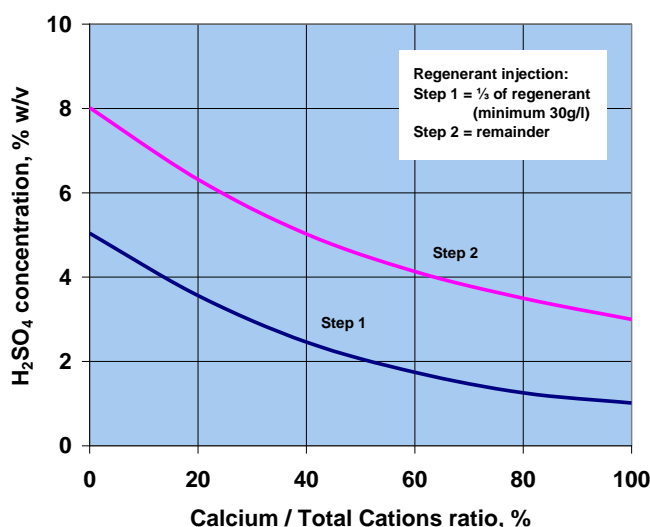
In counter-flow regeneration bed depths below 1000 mm (3 ft. 3 in) should be avoided and preferably beds in excess of 1200 mm (4 ft.) employed.

The regeneration level (amount of acid applied per litre or cubic foot of resin) will be lower than for co-flow regenerated units, typically between 60 and 120 g/l (4 – 7.5 lb/ft³). However, regeneration levels outside of this range are sometimes employed.

Sulfuric acid should be introduced step-wise at flow rates of 4 - 16 BV/h (0.5 - 2.0 gpm/ft³) and concentrations from 1 to 8% as indicated in Figure 2. The contact time between the resin and the regenerant solution should be between 20 and 40 minutes.

The slow (regenerant displacement) rinse is always carried out at flow rates similar to the acid injection step and in the same direction. This is to ensure a uniform contact time between the resin and the regenerant solution and that the rinse water follows the same route of the regenerant through the resin bed. Since slow rinse is usually more efficient in removing the spent regenerant from the resin than fast rinse, using more slow rinse can reduce the amount of final rinse required. Normally 1 to 2 BV (7.5 - 15 US gal/ft³) of slow rinse are adequate.

Figure 2 - Recommended maximum acid concentrations based on incoming calcium levels - Counter-flow regenerated columns



The final rinse is often carried out at the service flow rate. This also acts as a proving condition prior to returning to service after regeneration. Normally 2 - 4 BV (15 - 30 US gal/ft³) are required depending on the design of the distribution / collection system and the amount of slow rinsing previously performed.

It is more and more common in demineralization plants to employ closed-loop recycle rinses around the cation and anion units. This offers two advantages: it reduces the amount of waste water produced by the plant and it allows the design to include a proving pre-service rinse prior to placing the line back in service. Where anion resins sometimes develop long rinses due to organic fouling, a recycle rinse system can significantly reduce water consumption and avoid resins overloading.

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Table 2 - Typical Sulfuric Acid Regeneration conditions for counter-flow regenerated columns

Step	Design Basis	Duration
H₂SO₄ Injection	Refer to Figure 2 showing recommended acid concentration dependent on calcium level in the raw water.	Typically 20-40 minutes depending on regeneration level and flow rate
Slow Rinse	1-2 BV (7.5 to 15 gal/ft ³) at approx. regenerant flow rate	Typically 20-30 minutes depending on volume of water applied and flow rate
Final Rinse	2-4 BV (15 to 30 gal/ft ³) preferably at service flow rate or alternatively > 15 BV/h (2 gpm/ft ³)	Typically 10-20 minutes

(Key: BV = Bed Volume, BV/h = Bed Volume per hour)

Performance Data

The following graphs and correction factors are designed to help the design engineer to estimate the exchange capacity and hardness leakage achieved with Purolite C100 and Purolite C100H under different operating conditions. All the data shown are the result of years of industrial experience and are supplied in good faith. The final performance will depend on the detailed design and operation of the system, the quality of the regenerant chemicals as well as the long term maintenance of the plant. Some engineers who are using basic, standard plant of simple design may wish to take a design margin (safety factor) with regard to the published data to allow for less than ideal operation. Please note the data presented in this section are specific to co-flow regenerated designs with bed depths over 1000 mm (3 ft. 3 in) and counter-flow regenerated designs with bed depths over 1200 mm (6 ft. 6 in). For shallower bed depths there may be a requirement to down rate the expected performance depending on the quality of the design.

The data supplied are divided in three groups: figures 3 to 12 deal with capacity and leakage for co-flow regeneration, figures 13 to 20 with capacity and leakage for counter-flow regeneration and figures 21 to 22 with hydraulic data (backwash expansion and pressure drop). Within each of the first two groups there is a base capacity and a base leakage curve, both followed by other curves showing correction factors. To calculate the expected capacity or leakage, multiply the base capacity or leakage by the relevant correction factors.

For users interested in performing these engineering calculations electronically, Purolite's PureDesign™ software is available for download at www.purolite.com at no charge.

The data presented in this bulletin can also be used to estimate the operating performances of resins such as Purolite C100C, Purolite C100S or Purolite C100DL, while it is recommended to refer to dedicated engineering bulletins for products like Purolite PFC100 and Purolite PPC100.

Co-Flow Regeneration

Figure 3 provides base capacity data for Purolite C100, delivered in sodium form. Capacity of Purolite C100H, delivered in hydrogen form, is expected to be 8% lower than shown in this graph.

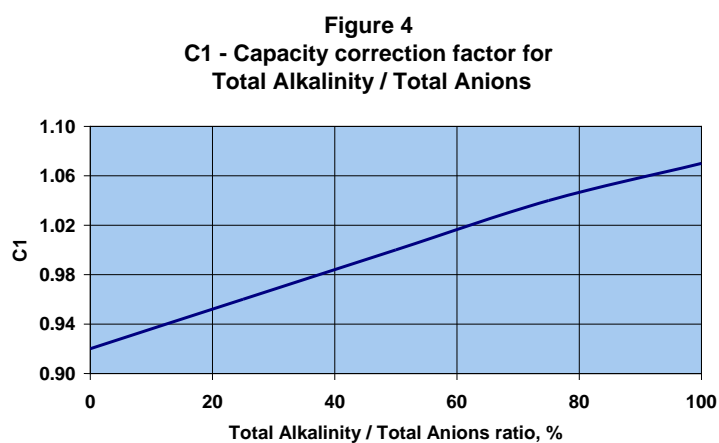
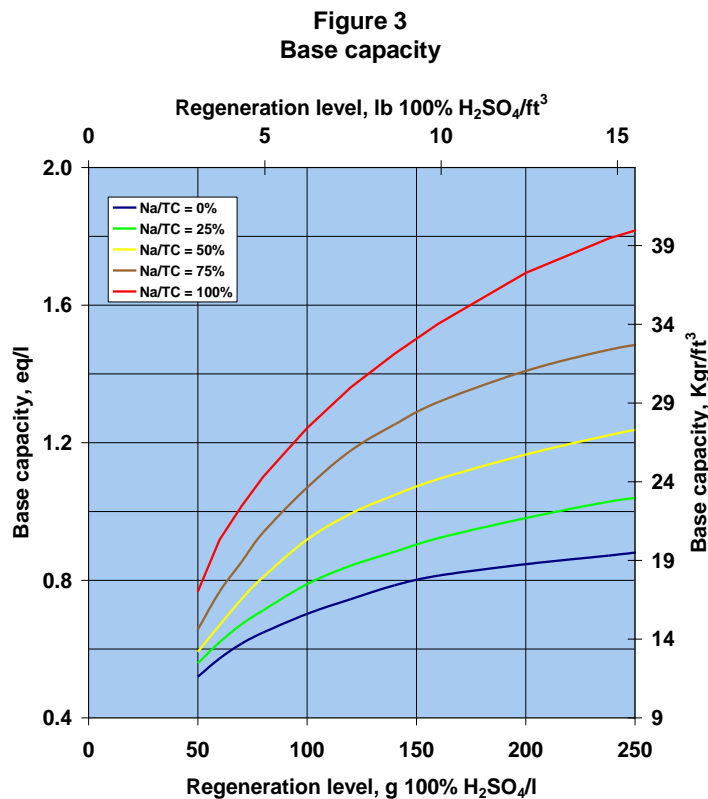


Figure 5
C2 - Capacity correction factor for temperature

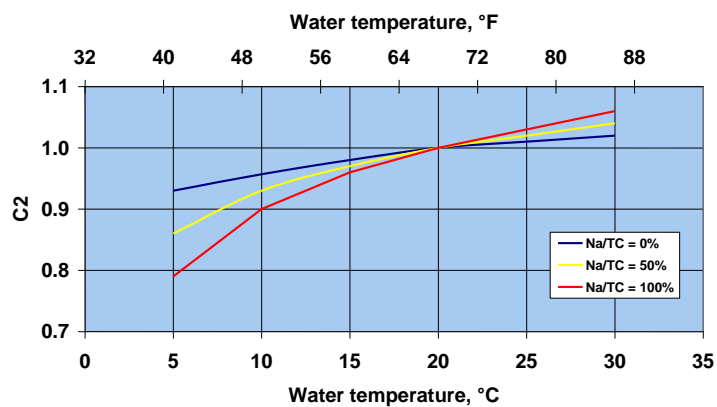


Figure 6
C3 - Capacity correction factor for cycle time

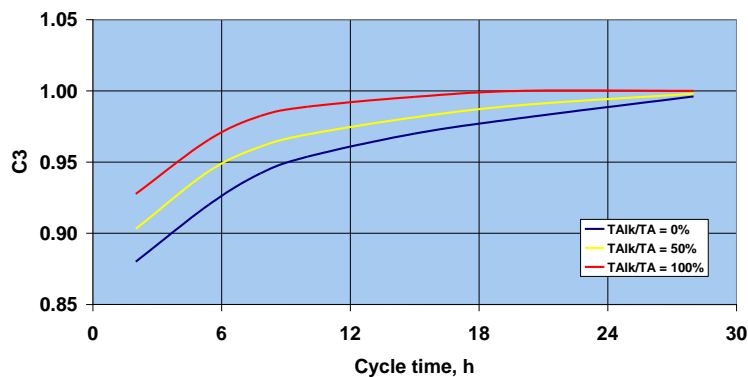


Figure 7
C4a - Capacity correction factor when Na / Total Cations = 0%

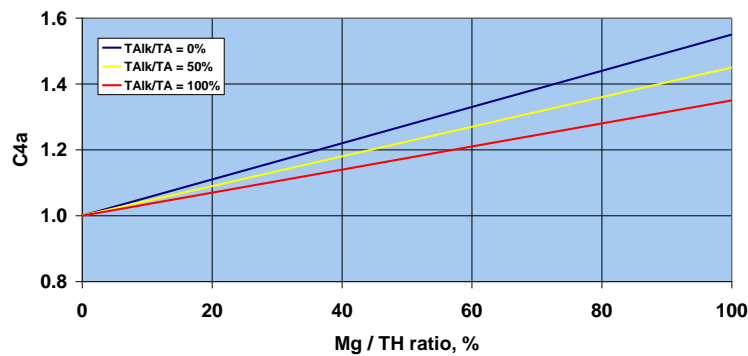


Figure 8
C4b - Capacity correction factor
 when Na / Total Cations = 50%

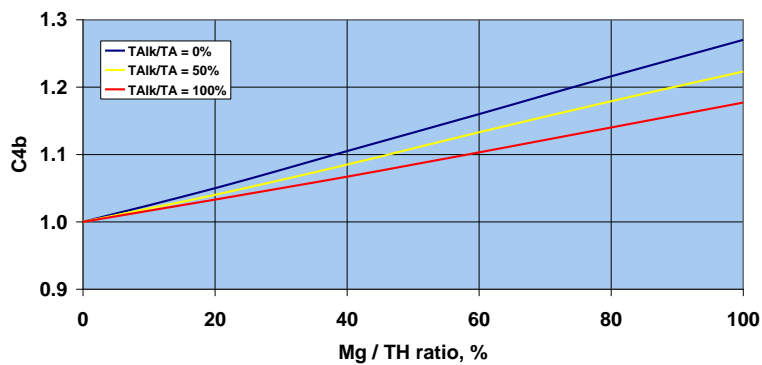


Figure 9
C4c - Capacity correction factor
 when Na / Total Cations = 100%

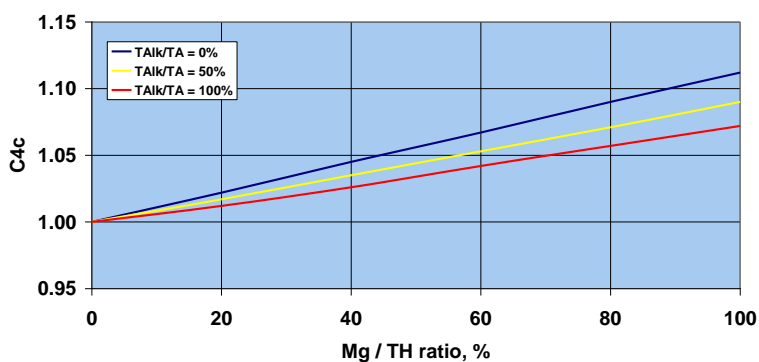
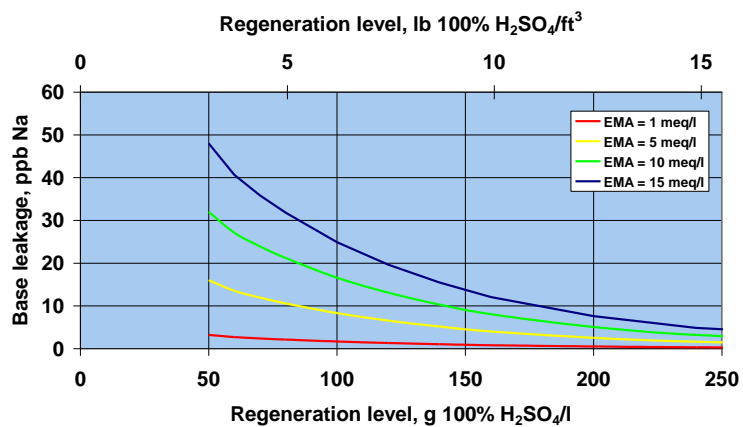


Figure 10
Base Sodium Leakage



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Figure 11
L1 - Leakage correction factor for
Total Alkalinity / Total Anions

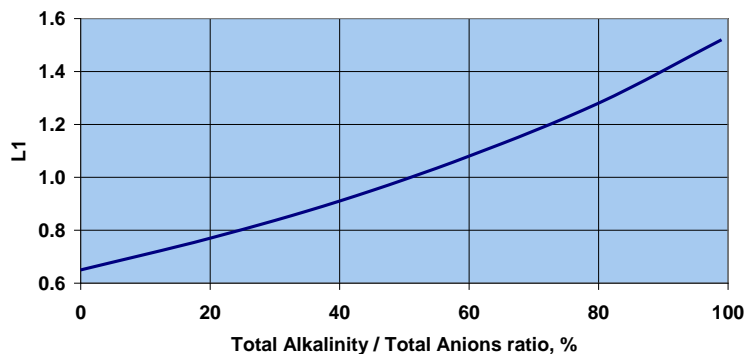
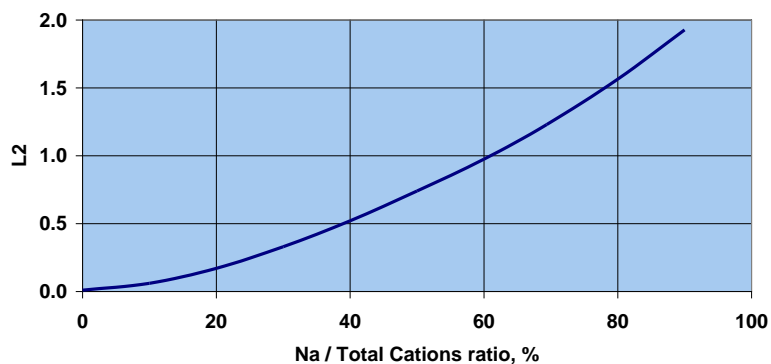


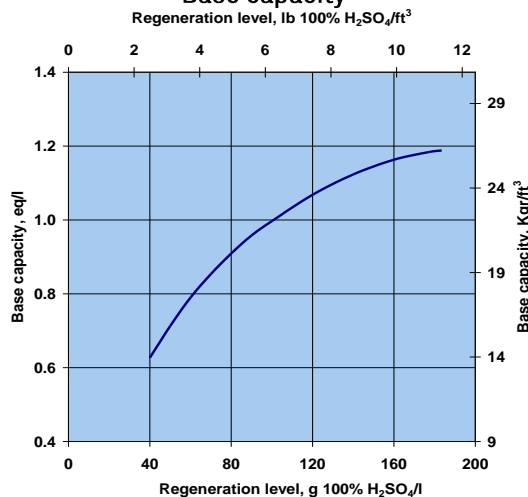
Figure 12
L2 - Leakage correction factor for
Na / Total Cations



Counter-Flow Regeneration

Figure 13 provides base capacity data for Purolite C100, delivered in sodium form. Capacity of Purolite C100H, delivered in hydrogen form, is expected to be 8% lower than shown in this graph.

Figure 13
Base capacity



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Figure 14
C1 - Capacity correction factor for Na / Total Cations

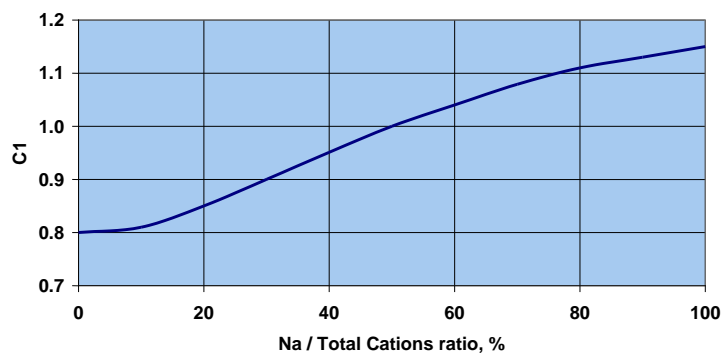


Figure 15
C2 - Capacity correction factor for
Total Alkalinity / Total Anions

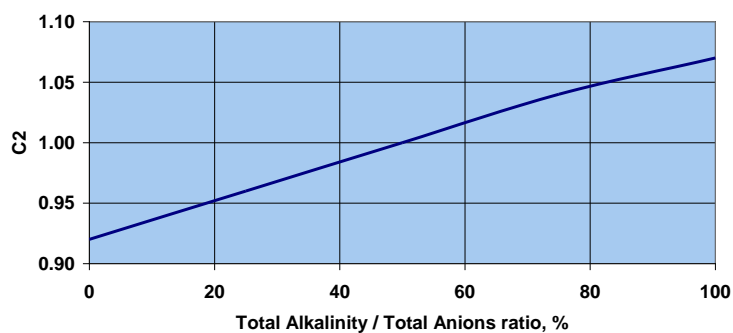


Figure 16
C3 - Capacity correction factor for temperature

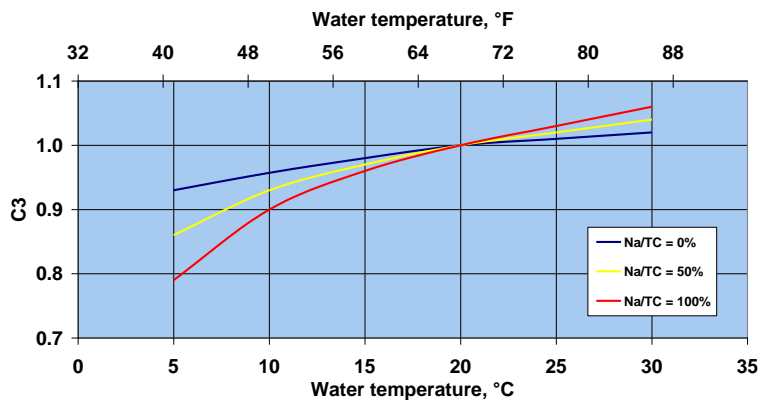


Figure 17
C4 - Capacity correction factor for cycle time

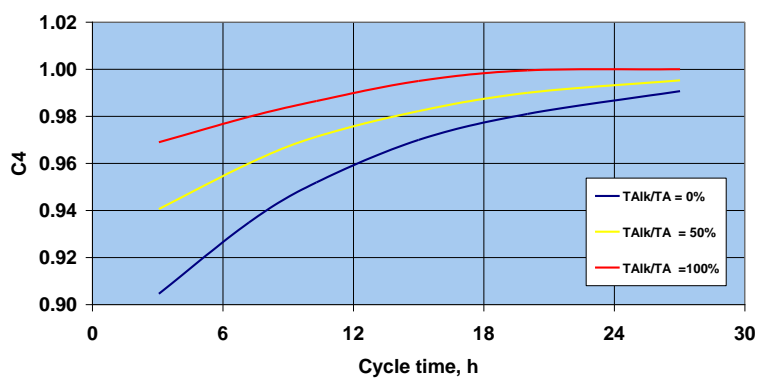


Figure 18
C5 - Capacity correction factor for bed depth

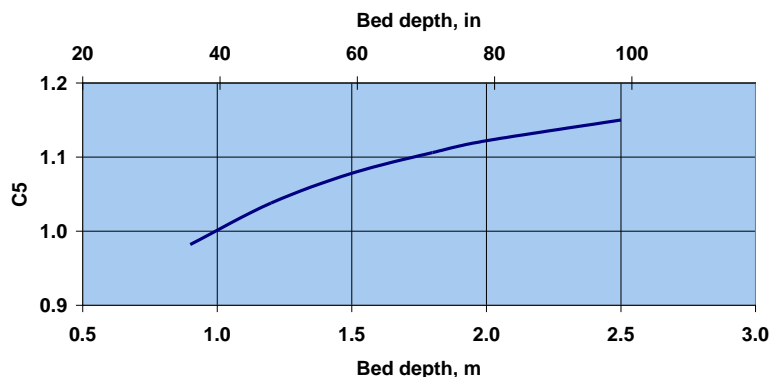


Figure 19
C6 - Capacity correction factor for specific flow rate

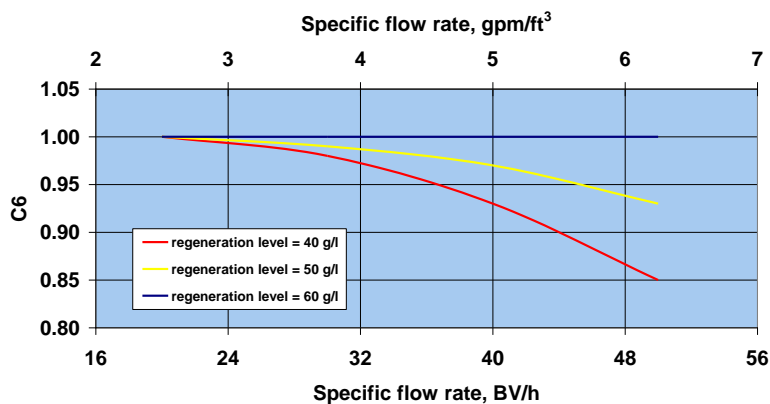
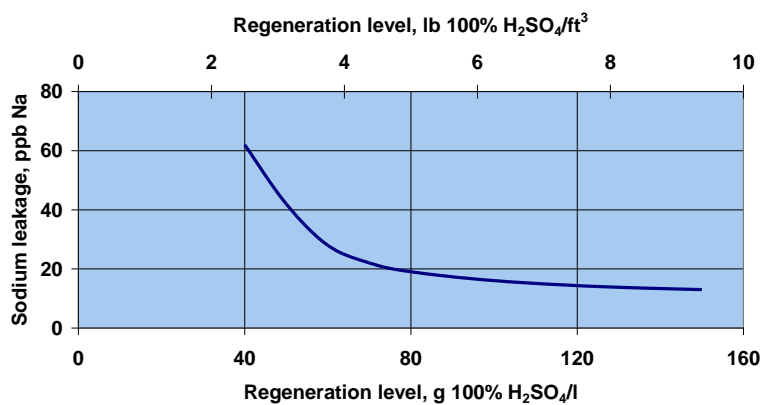


Figure 20
 Sodium leakage



Hydraulic Characteristics

Figure 21
 Backwash expansion

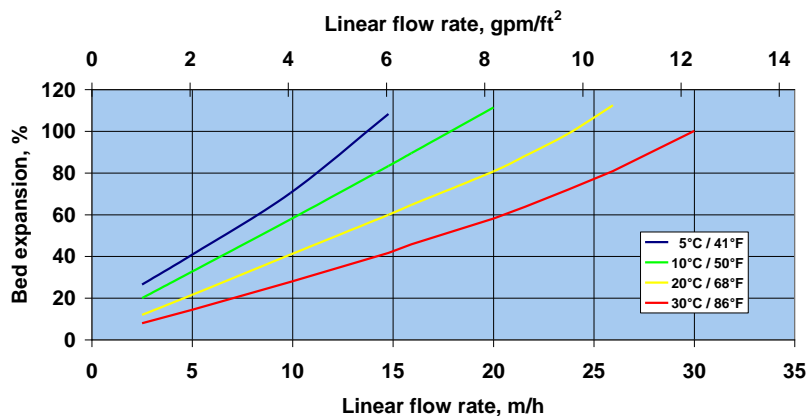
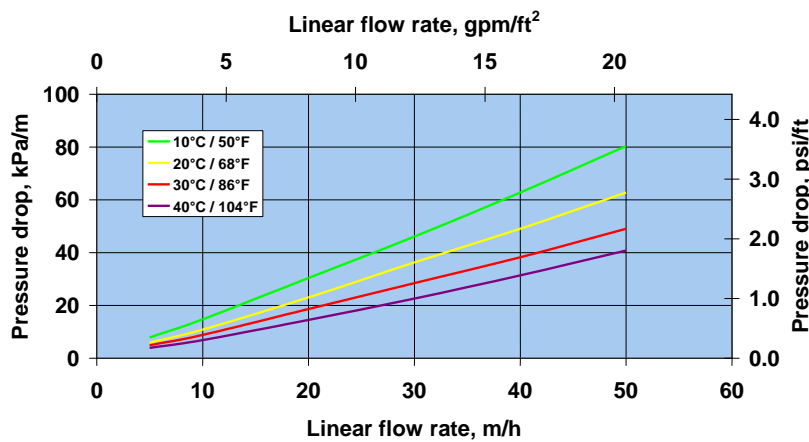


Figure 22
 Pressure drop



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Additional information & application notes

Safety: Strong oxidants, such as nitric acid, may cause violent reactions with ion exchange resins under certain conditions. Use of strong oxidants must be done under the care and supervision of persons knowledgeable in handling these types of materials.

Notice: The information contained in this document, including application, technical specifications and operation utilizing Purolite products is provided in good faith and without warranty. Use of our products is out of our control. It is important that you test our products for suitability in your application and environment. Testing should include health, safety and material handling as well as application. Unless otherwise agreed, all products are sold according to our General Terms and Conditions of sale. It is expressly understood that you undertake all responsibility and liability for use of the product. No license is implied or in fact granted under the claims of any patent. No other warranty of any kind, express or implied, whether of fitness or merchantability is made as to the material sold or any instructions or technical advice provided, unless separately agreed in writing.

Americas

150 Monument Road
Bala Cynwyd, PA
19004
T +01 800.343.1500
T +01 610.668.9090
F +01 484.384.2751
Americas@purolite.com

Europe

Llantrisant Business Park
Llantrisant
Wales, UK
CF72 8LF
T +44 1443 229334
F +44 1443 227073
Europe@purolite.com

Asia Pacific

Room 707, C Section
Huanglong Century Plaza
No.3 Hangda Road
Hangzhou, Zhejiang, China 310007
T +86 571 876 31382
F +86 571 876 31385
AsiaPacific@purolite.com



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