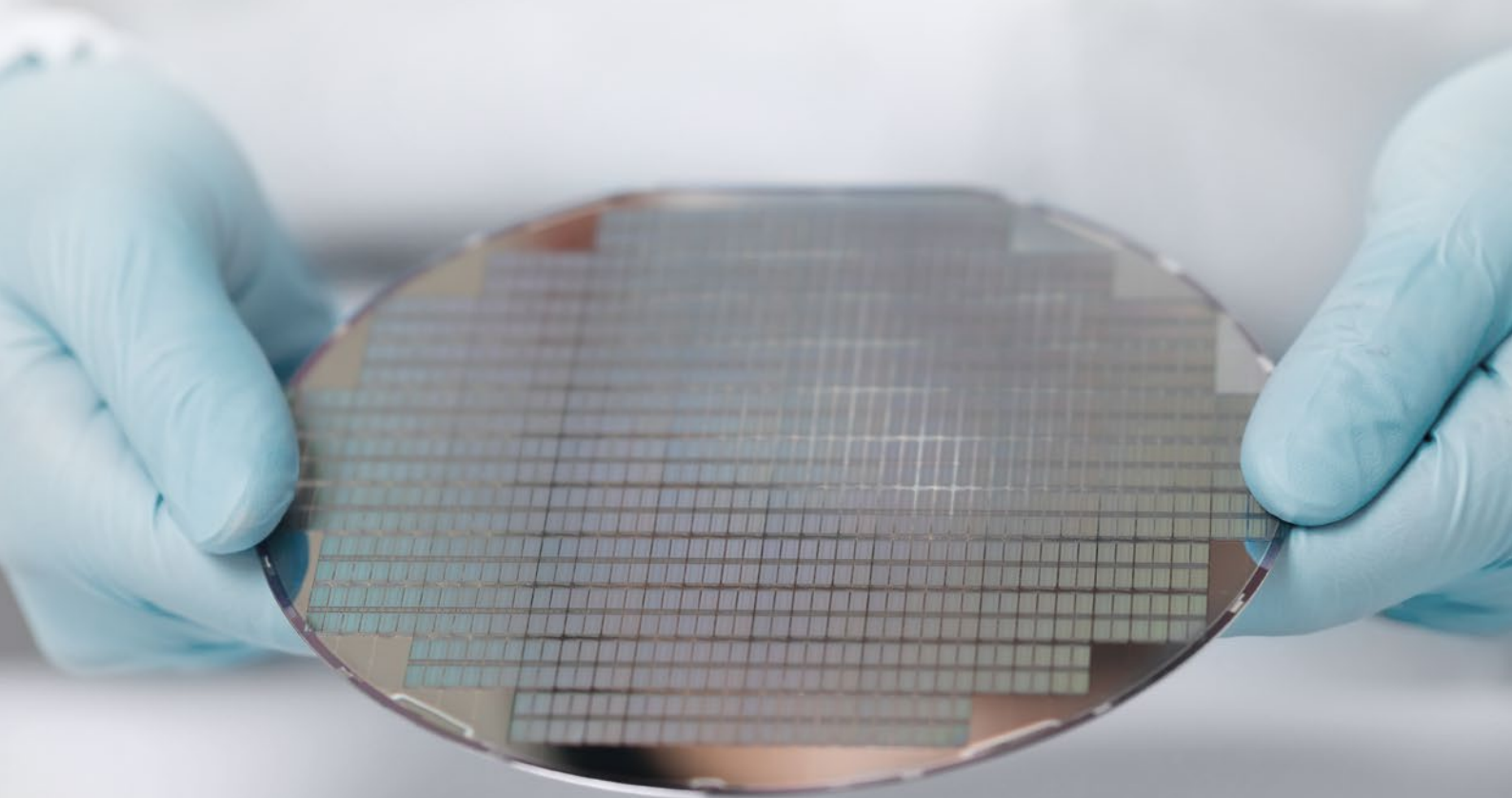


# QUALITY PURIFIES.



Refined ion exchange resins for **ultrapure water** applications.

**X** Lewatit®

**QUALITY WORKS.**

**LANXESS**  
Energizing Chemistry



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# LET OUR COMPETENCE AND EXPERIENCE ASSIST YOU



## LANXESS – Your global partner for success

We are a leading specialty chemicals company based in Cologne, Germany, well established on the global market. Our primary expertise lies in producing, developing, and marketing chemical intermediates, additives, specialty chemicals, and plastics.

We manage our operating business through four segments: Advanced Intermediates, Specialty Additives, Consumer Protection, and Engineering Materials. The business units within these segments work with a broad range of applications and markets. As a specialist and efficient partner, we offer solutions to all kinds of challenges faced by our customers. We focus on our customers' requirements in order to drive progress and reliably provide innovative product, material and service solutions. Our manufacturing, administration, and logistics processes are designed for efficiency and performance.

The Liquid Purification Technologies (LPT) business unit offers a broad range of technologies and solutions for the treatment of water and other liquid media. It is one of the leading manufacturers of ion exchange resins with production sites in Germany and India. Its excellent technical know-how and extensive customer service, based on many decades of experience, are highly acknowledged. In addition, LPT also offers a range of Bayoxide® iron oxide adsorbers for various water treatment applications.

Additionally, our unique calculation and design software LewaPlus® allows for planning of ion exchange, reverse osmosis, and ultrafiltration systems, and combinations thereof under various system configurations, including process configurations only available with Lewatit® product technology.

## Lewatit® – Our valued brand

For more than 80 years our Lewatit® ion exchange resins and adsorbers have been employed in numerous industries to purify, treat, and remediate water and other liquid media. Important areas of application include:

- Chemical and petrochemical
- Food and beverages
- Household
- Mining and metallurgy
- Municipal water treatment
- Paper and pulp
- Pharma and biotech
- Power generation
- Semiconductor and photovoltaic.

Our products and solutions tailored to produce ultrapure water for use in the semiconductor and photovoltaic industry are presented in this brochure.

# ULTRAPURE WATER – WORLDWIDE AND TO SUIT EVERY REQUIREMENT

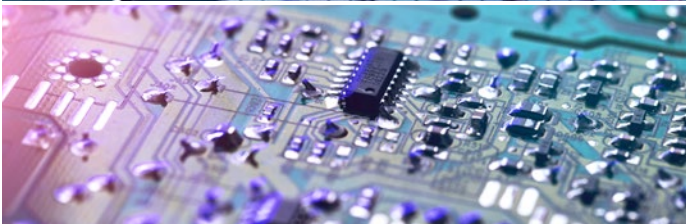
Activities in many areas of industry, research, and development depend on water that has to meet very particular purity and quality requirements. This includes fully desalinated or de-ionized (DI) water with a specific electrical conductivity within a range of 0.1–1  $\mu\text{S}/\text{cm}$  as well as ultrapure water (UPW) with a specific electrical conductivity of approximately 0.055  $\mu\text{S}/\text{cm}$  and electrical resistivity of roughly 18.2  $\text{M}\Omega\cdot\text{cm}$  at 20°C. The latter, for example, is required in the complex processes of semiconductor production. The pharmaceutical industry, medical field, and aerospace sector also use ultrapure water.

## Ultrapure water applications



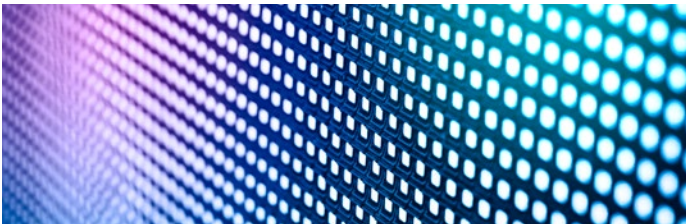
### Materials

- Wafer
- Glass substrate



### Semiconductors

- Front end processing
- Back end processing



### Display panels

- Thin-film transistor liquid crystal display panel (TFT-LCD)
- Active matrix organic light emitting diode panel (AM-OLED)



### Solar panel production

- Roof panels

Particularly when it comes to the manufacture of semiconductor devices, which include microchips, LCD and OLED displays, as well as photovoltaic cells, the requirements concerning the purity of the water used has become more and more demanding with each new generation of semiconductor devices. The smaller the dimensions of the electronic assemblies, the more sensitive the production processes are

to foreign ions and dissolved organic impurities in water but also to the presence of micro- and nanoparticles. The quality of the feed water used plays a crucial role in terms of organic impurities, with ultrapure water systems often having to be custom-designed to suit the local conditions. There may be a need for additional filtration or adsorption stages to remove the natural organic matter (NOM) contained in untreated water.

The SEMI F63 (Guide for Ultrapure Water Used in Semiconductor Processing) standards issued by SEMI are a key source of guidance with regard to these requirements.

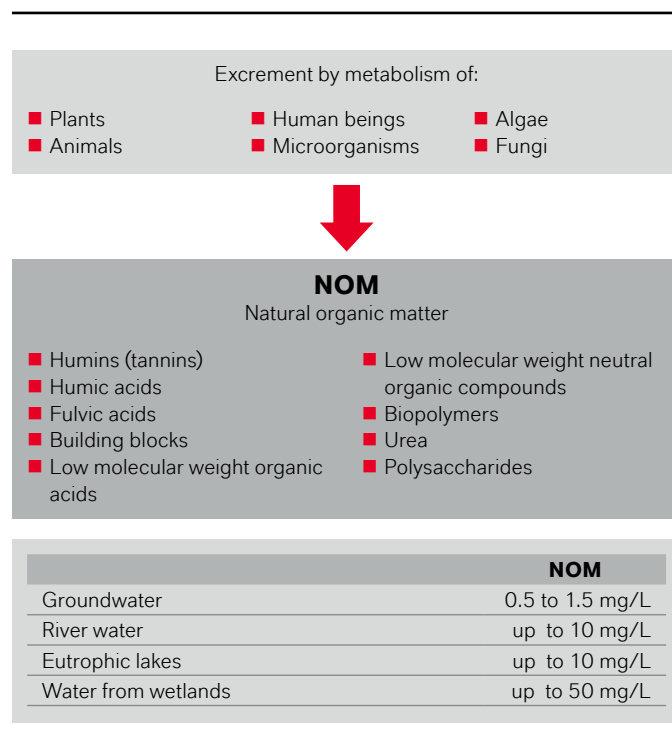
Complying with these standards is our stated aim and this gives our customers a high degree of confidence in utilizing our ion exchange resins solutions in their high-purity water systems. The International Society for Pharmaceutical Engineering (ISPE) defines similar guidelines for laboratories and companies in the chemical and pharmaceutical industries.

**Key quality criteria for ultrapure water ion-exchange resins:**

- Mechanical and osmotic stability of the resin beads
- Achievable specific conductivity
- Operating capacity
- Ions, particles, and organic matter or total organic carbon (TOC) released into the water
- Metal content of resin matrix and release of metal (ions)
- Separability of resin components in the case of regenerable mixed beds
- Kinetics of ion exchange

LANXESS takes up these challenges in collaboration with users and many providers of systems for producing ultrapure water. We are constantly evaluating, enhancing, and extending our product line of ultrapure ion-exchange resins. We work with system manufacturers on process improvements and with analytics specialists on ensuring that we can reliably identify and remove traces of impurities to the ppb and ppt levels as well as nanoparticles, often close to the limits of detection.

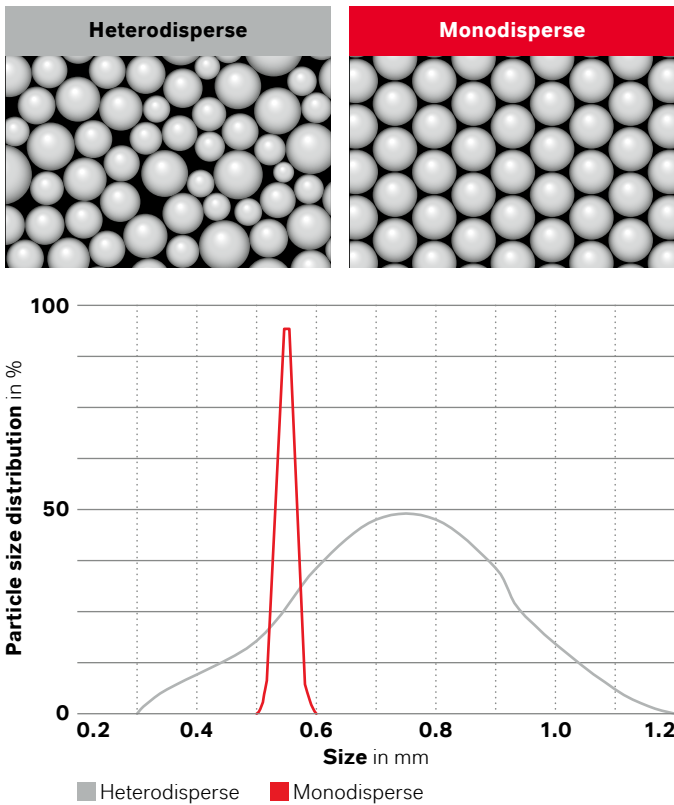
**Figure 1: Influence of natural organic matter (NOM) for the production of ultrapure water**



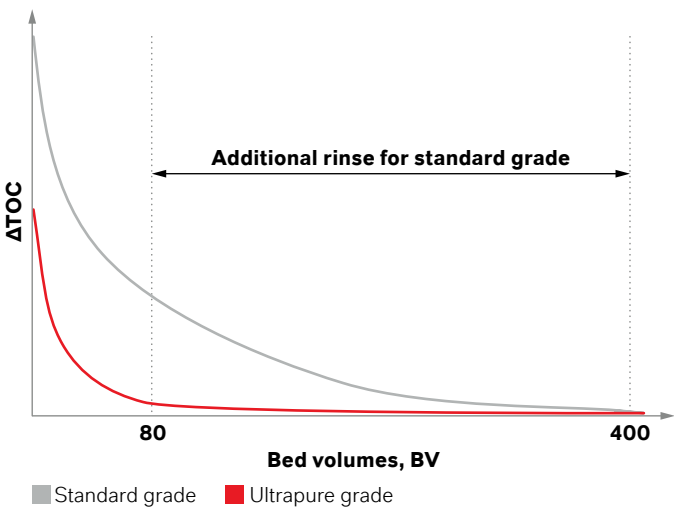
Mika Sillanpää, Natural Organic Matter in Water – Characterization and Treatment Methods, Butterworth-Heinemann, Oxford 2015

# LEWATIT® ULTRAPURE – EXACTLY WHAT YOU NEED

**Figure 2: Monodisperse vs. heterodisperse ion exchange resins**



**Figure 3: Rinsing properties after resin installation: ultrapure grades compared to standard grades**



## The right resins for every type of system

In addition to industrial-scale ultrapure water production facilities, compact package units, as they are known, are playing an increasingly important role particularly for laboratories and small production plants in the electronics industry. The former usually have regeneration units for the ion-exchange resins used. Final polishing usually involves the use of non-regenerable "single-use" resins. The situation is different in the case of package units. These use cartridges filled with ion-exchange resins, which are exceptionally easy to use, enabling extremely reliable system operation. Such cartridges are intended to be used only once, so the resins are not regenerated. LANXESS is equally familiar with both methods. We offer regenerable and single-use resins for large-scale facilities, as well as special resins, especially mixed-bed resins, for filling cartridges. Users all around the world, in laboratories and in industry, have been confidently relying on the consistently high quality of ion-exchange resins from the Lewatit® UltraPure product range for many years.

## Our formula for success: a consistent bead size

With regard to ultrapure water resins, we generally use resins with narrow polymer bead size distribution, known as monodisperse resins. For this purpose, we have dependable, high-performance processes, state-of-the-art production facilities, and many years of experience at our disposal. All of this offers various practical advantages over ion exchangers with wide bead size distribution.

## Monodisperse resins – The advantages summed up:

- More stable when exposed to mechanical and osmotic stresses
- Lower pressure loss in operation
- Higher operating capacity
- Faster exchange kinetics

For end users, this means long useful lives for the exchange resins, maximum operational reliability, and less need for maintenance.

## Lewatit® UltraPure resins – The benefits in detail

### Short start-up phase following resin change

After lengthy operation, the exchange resins in an ultrapure water production facility have been optimally rinsed with clean water as a result of extended loading times and give off almost no more impurities or particles.

Minute impurities, which are inevitable in brand-new resins, require a start-up phase after any resin change before ultrapure water can again be produced to the expected standard of quality. The shortest possible start-up phase is preferable to ensure, for example, that semiconductor production can be resumed as quickly as possible without impairing quality. We take this requirement into account by means of a special morphology for the polymer beads of our ultrapure resins, which enables any impurities in the liquid phase to be diffused swiftly and efficiently. A comparison of the  $\Delta$ TOC values in water for a standard resin and an ultrapure resin demonstrates this.

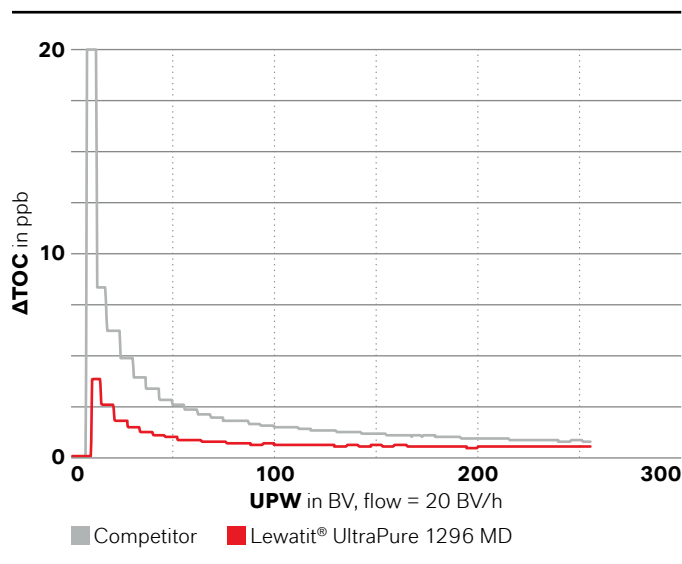
In the case of Lewatit® UltraPure 1296 MD, used in final polishing, a  $\Delta$ TOC value of approximately 0.4 ppb is reached downstream of the exchange stage after a start-up phase of around just 45 bed volumes or roughly two hours.

### Specification-compliant metal content

The International Technology Roadmap for Semiconductors (ITRS) presents and forecasts ambitious requirements for ultrapure water in terms of the content of various ions, including those of numerous light and heavy metals. Some of these requirements are approaching the limit of detection of the analytical methods currently available in the ppb/ppt ranges.

Final polishing resins from LANXESS, such as Lewatit® UltraPure 1296 MD, already fulfill many of these requirements. In some cases, however, special downstream cleaning processes are required. Furthermore, insufficiently standardized measurement methods do not always allow readings obtained through different means to be compared directly.

**Figure 4: UPW rinse test: Lewatit® UltraPure 1296 MD versus competitor polisher**



**Table 1: Ion release below detection limit, example Lewatit® UltraPure 1296 MD**

Detection limits are constantly being adjusted		Detection Limit in ppt
<b>Ion Chromatography</b>		
Li <sup>+</sup> , Na <sup>+</sup> , Mg <sup>2+</sup>		0.2
K <sup>+</sup> , Ca <sup>2+</sup>		1
NH <sub>4</sub> <sup>+</sup> , TMA		5
F <sup>-</sup>		3
Cl <sup>-</sup> , Br <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> , NO <sub>3</sub> <sup>-</sup>		5
PO <sub>4</sub> <sup>3-</sup> , SO <sub>4</sub> <sup>2-</sup>		20
<b>Inductively Coupled Plasma Mass Spectrometry</b>		
Ba, Be, Cd, Co, Cu, Cr, Ga, Mn, Ni, Pb, Sr, V, Zn		0.5
Ag, Al, As, Bi, Ce, Cs, Dy, Fe, Er, Eu, Gd, Ge, Hf, Ho, In, Ir, La, Mo, Nb, Nd, Os, Pd, Pr, Rb, Re, Ru, Sb, Sm, Ta, Tb, Te, Tl, Th, Tm, Ti, U, W, Y, Yb		1
Au, Pt, Rh, Sc, Sn, Zr		2
Hg		5
B		10
Se		100
<b>Dissolved Silica</b>		
		100

# METHODS OF PRODUCING ULTRAPURE WATER

The efficient and application-specific production of ultrapure water with ion exchangers requires seamless interaction between resin quality, process engineering, and system design. The processes are centered around ion exchange. Following pre-cleaning of the feed water, usually with filters or activated carbon, ion-exchange resins play an important role in many cases, possibly combined with reverse osmosis, during the primary desalination stage. Because LANXESS also offers such resins, this makes it possible for this desalination step to be ideally matched to the subsequent final polishing. Combinations with electrodeionization (EDI) or ultra-/nanofiltration stages for removing particles are also widely used. The latter are becoming all the more significant as the requirements concerning tolerable particle concentrations and sizes in ultrapure water become ever more stringent.

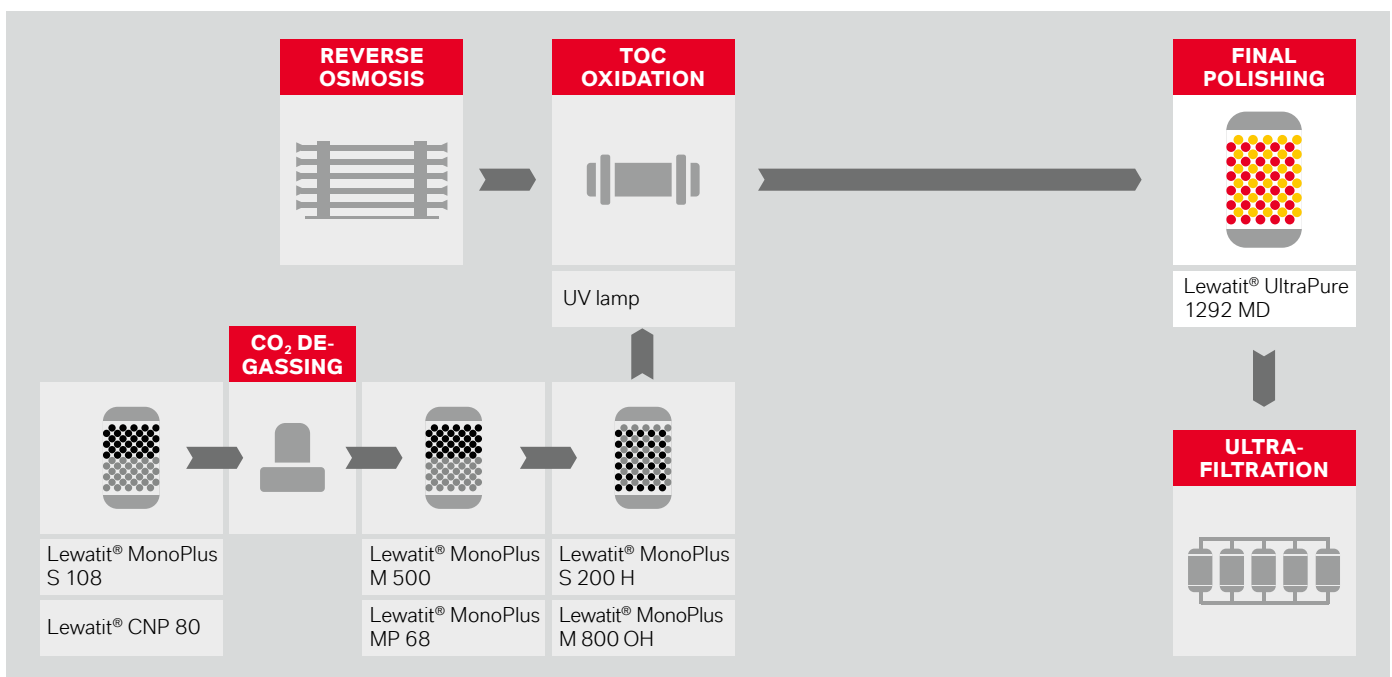
The basic design of such systems depends on the area of application of the water. The manufacture of photovoltaic cells, for example, typically requires water with a resistivity of more than 18.0 MΩ\*cm and a TOC figure of less than 50 ppb. This is obtained after pre-cleaning through a multi-stage ion-exchange process or through reverse osmosis as

well as subsequent removal of organic impurities by means of UV irradiation in the presence of oxygen. Final polishing takes place in a mixed-bed exchanger, possibly one that is regenerable (Lewatit® UltraPure 1292 MD), followed by ultra-filtration to reduce particle concentration. Particles as small as 50–100 nm in diameter are removed in this process, as measurements using conventional analytical methods (Ultra DI® 50 from Particle Measuring Systems (PMS), a company based in Darmstadt) have demonstrated.

Final polishing of ultrapure water required for high-end applications, such as the production of microchips, is more complicated. In addition to a resistivity in the range of the inherent resistivity of pure water, a TOC value of less than 1 ppb is required in some cases, as well as an ever lower concentration of particles with diameters of 10 nm or less.

The formation of hydrogen peroxide in photochemically induced radical reactions during UV treatment is particularly worthy of attention in this context (for example, see M.P. Herrling, P. Rychen, *Ultrapure Micro Journal* 1 (2019) 1, 34–43). Hydrogen peroxide has considerable potential for

**Figure 5: Example of ultrapure water production for solar panels**



Usual water specifications: >18.0 MΩ\*cm, <50 ppb TOC

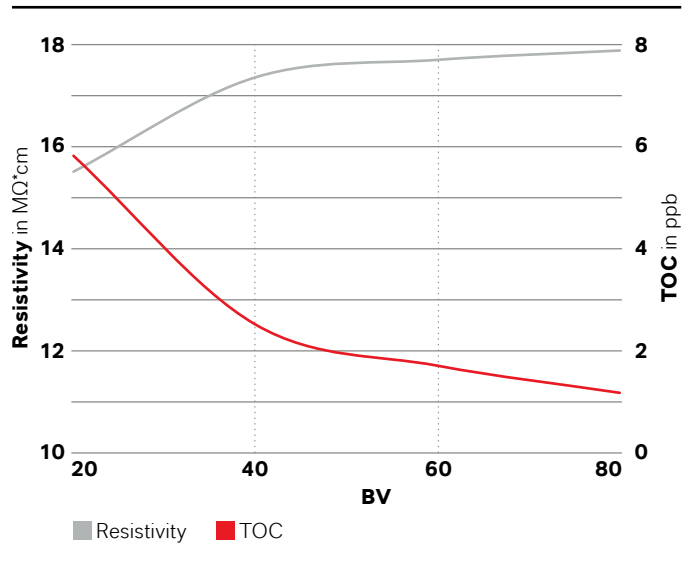


breaking down ion-exchange resin beads in the subsequent ion-exchange units, resulting in increased formation of particles smaller than 10 nm. Such particles, which can currently be analyzed only with considerable difficulty, if at all, present a major challenge in the manufacture of the latest generations of integrated circuits in particular; for example, using 10 nm, 7 nm, or even 5 nm technology.

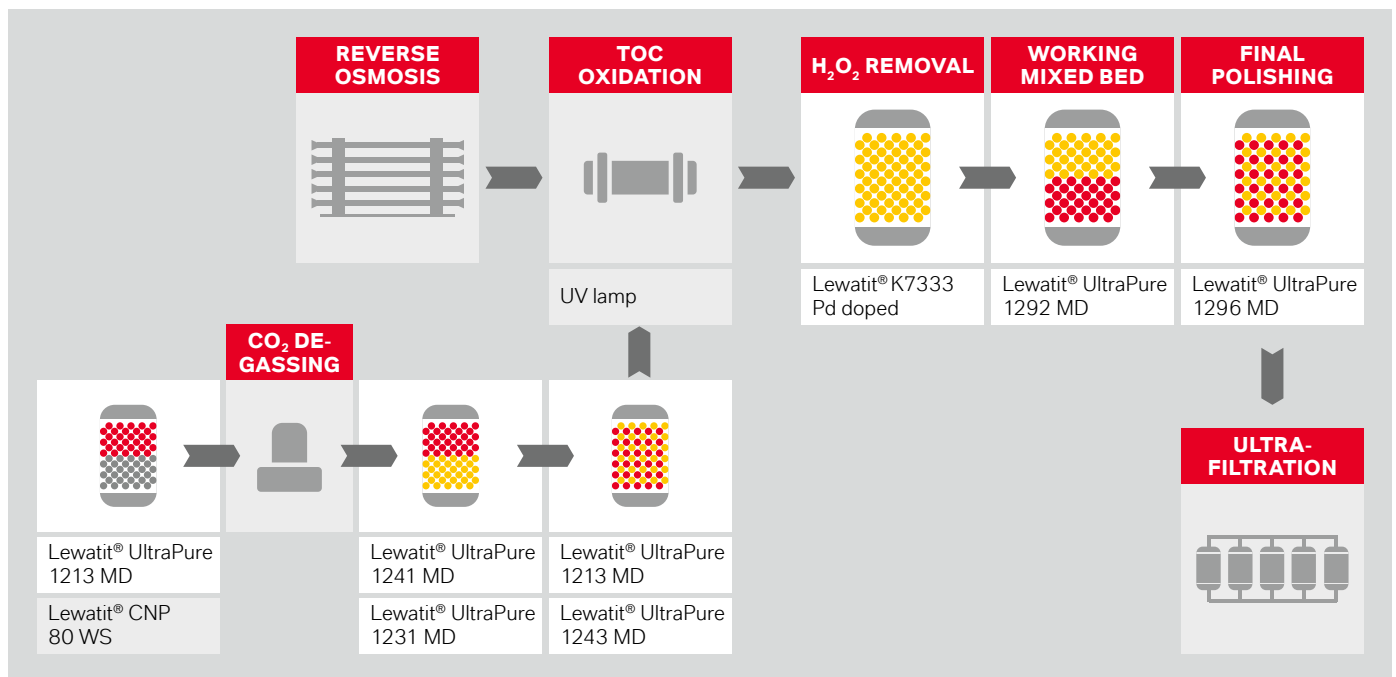
LANXESS has devised an innovative solution to this problem. This involves employing Lewatit® K 7333 palladium-doped exchange resin. Hydrogen peroxide that has formed is largely or entirely broken down in this process. The subsequent working mixed bed and final polishing are thus efficiently protected from H<sub>2</sub>O<sub>2</sub>-induced erosion. Ultra- or nanofiltration finishes off the production process in this case as well. Measurements have shown that Lewatit® K 7333 does not adversely affect resistivity nor the TOC figure after a brief start-up phase.

LANXESS works with renowned analytical specialists to ensure that it is able to analyze and assess particularly those concentrations of particles in the sub-10 nm range identified as critical.

**Figure 6: Rinsing properties of Lewatit® K 7333**



**Figure 7: Example of ultrapure water production for semiconductor applications**



Usual water specifications: >18.2 MQ·cm, <1.0 ppb TOC

# LEWATIT® ULTRAPURE MAKES THE DIFFERENCE

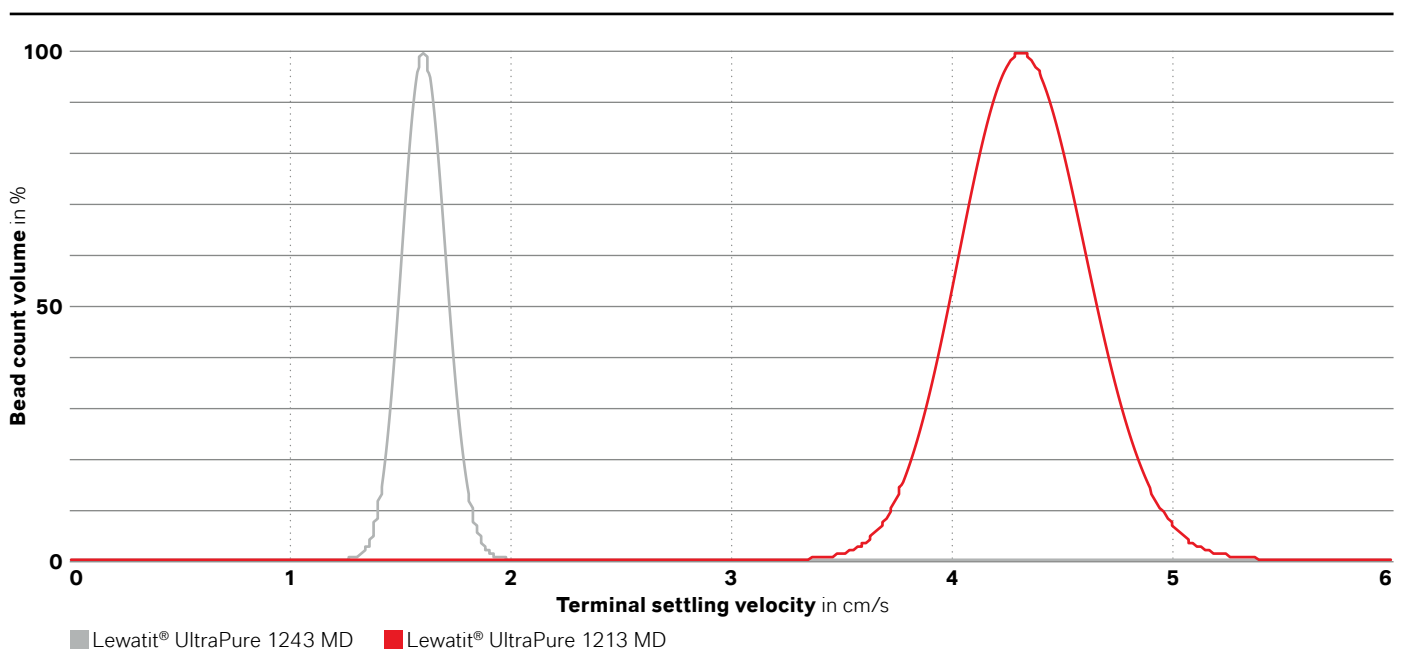
## Tailor-made ready-to-use mixed beds

The resin components used in the UltraPure ready-to-use mixed beds have properties optimized for specific applications thanks to a special polymer matrix containing resin beads with a customized diameter. This allows us to offer both working mixed beds with regeneration (Lewatit® UltraPure 1292/94 MD) and without regeneration (Lewatit® UltraPure 1297 MD) as well as final polishing resins (Lewatit® UltraPure 1294/96 MD), which are also non-regenerable and provide ultrapure water of an exceptionally high standard of quality.

Strongly acidic cation-exchange (SAC) resins always have higher densities than strongly basic anion-exchange (SBA) resins. In the case of working mixed beds with regeneration, one must separate the resins in the column for regeneration by means of backwashing with DI water. Separation should ideally be complete, which is identifiable by a distinct separating line between differently colored resins.

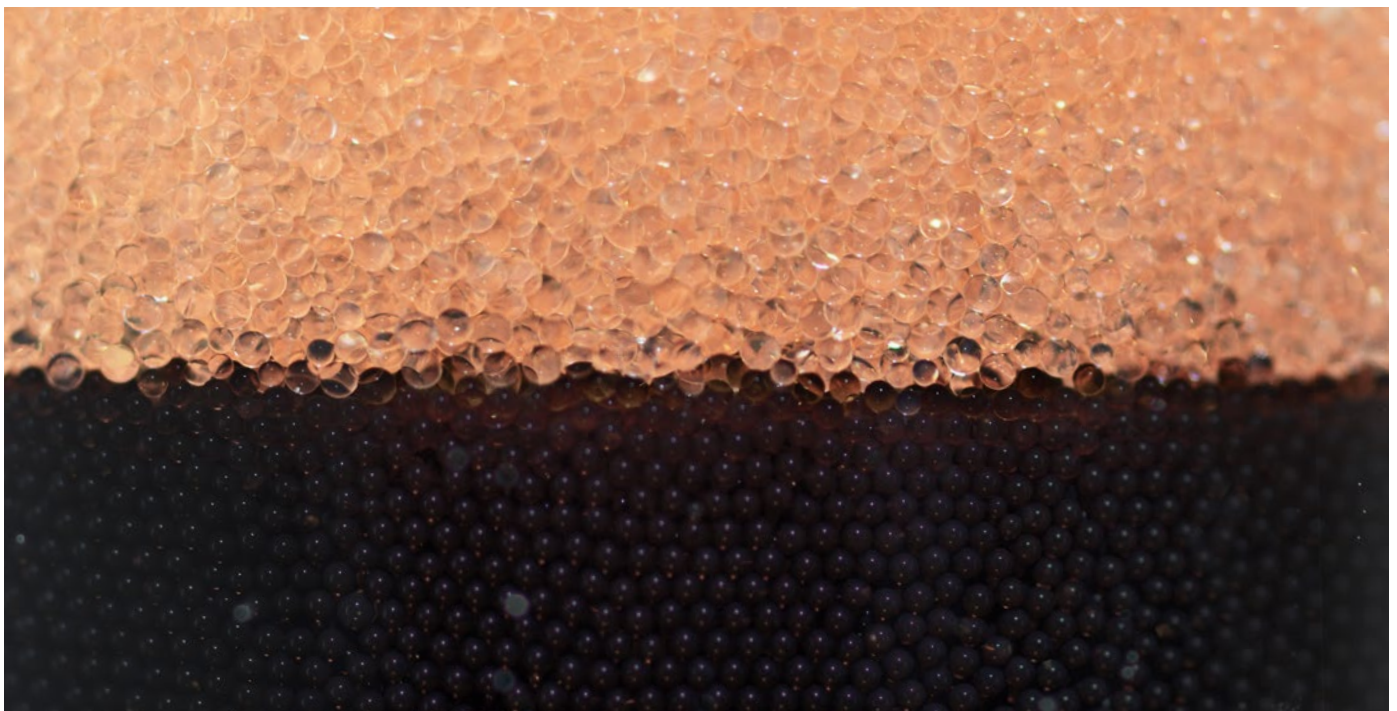
If the SAC beads are smaller than the SBA beads, this has a detrimental effect, because it partially or entirely offsets

**Figure 8: Perfect resin separation and regeneration through monodisperse technology**



the effect of the higher density. Distinct separation is then no longer possible, and SAC beads remain in the SBA resin layer. During regeneration with sodium hydroxide, these small SAC beads are converted to the sodium form and cause a loss in performance (sodium slippage) on the part of the working mixed bed. Monodisperse resins, with their narrow bead size distribution and similar bead size for both components, guarantee complete separation along a distinct separating line and thus optimum performance.

The breakthrough of a single, weakly bound type of ion may require regeneration of a working mixed bed that is only partially doped and has an SBA resin that is still doped with an appreciable number of OH<sup>-</sup> ions. To achieve this, depending on the application, it is possible either to convert the SAC to the sodium form or to use a suitable surfactant that reduces ionic interactions between SBA and SAC resins that would prevent complete separation. Special surfactants, such as polymeric alkylammonium salts, have proved themselves especially well suited for this purpose. They can be rinsed out quickly and completely during the start-up phase after regeneration. Separating the resin is generally not necessary at the final polishing stage because it is not regenerated.



■ Optimal separation of SBA resin (Lewatit® UltraPure 1243 MD, light) and SAC resin (Lewatit® UltraPure 1243 MD, dark)

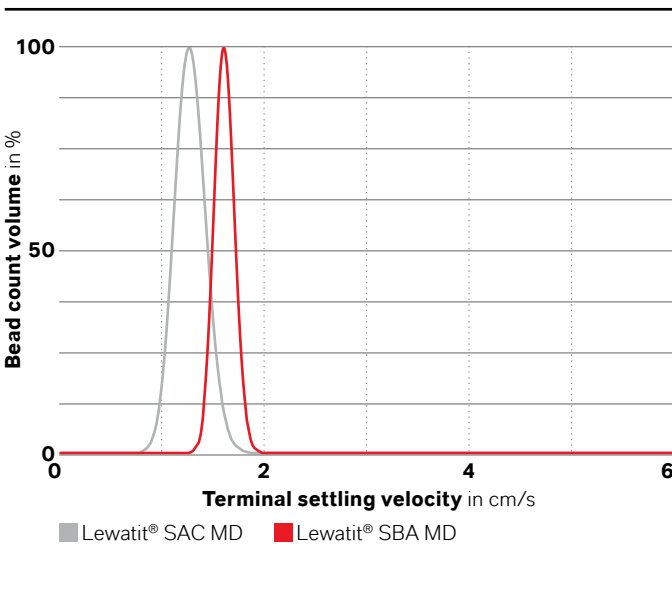


■ Resin stability control and bead size measurement

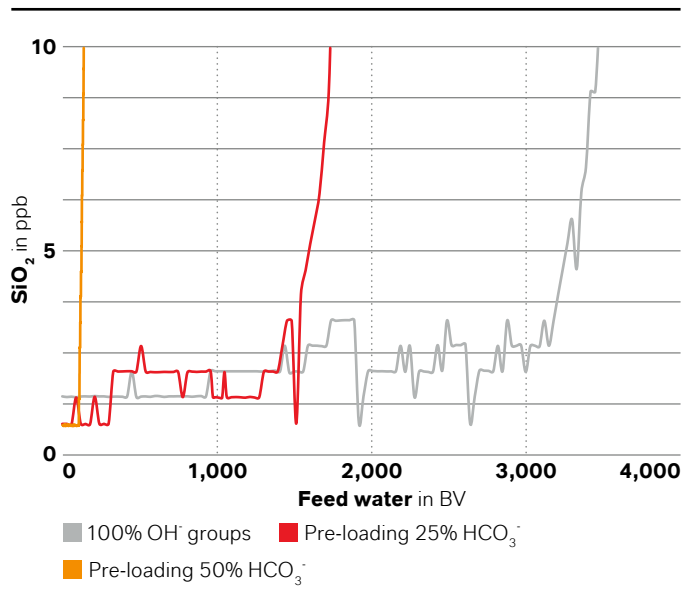
In the Lewatit® UltraPure 1296 MD ready-to-use mixed bed, the beads of the SAC component are deliberately somewhat smaller than those of the SBA component so as to ensure a permanently stable, homogeneous mixture, and thus an optimized final polishing process. Another benefit of this variant is improved cation kinetics, which results in efficient cation retention even at high specific and metric filtration rates.

If separating both mixed-bed components is not necessary or desirable, e.g., in cartridges, reducing the SAC component further prevents any gradual vibration-induced demixing from occurring. Lewatit® UltraPure 1297 MD is ideal for cartridges and other single-use applications because the components are practically inseparable, which also means that they are not regenerable, however.

**Figure 9: Less separable mixed bed Lewatit® UltraPure 1297 MD**



**Figure 10: Wrong storage causes reduced silica capacity because of CO<sub>2</sub> contamination**



**Optimized silicate and borate removal**

Ultrapure resins used in the production of ultrapure water contain highly regenerated resin components. Except for trace amounts, the cation exchangers are in the hydrogen form while the anion exchangers are almost entirely in the hydroxide form.

A highly regenerated anion exchanger of this type adsorbs carbon dioxide from the air, which means that the hydroxide ions bound to the functional groups accumulate CO<sub>2</sub> and thus become hydrogen carbonate ions (HCO<sub>3</sub><sup>-</sup>). HCO<sub>3</sub><sup>-</sup>-doped functional groups of the exchange resin are no longer available for absorbing silicate or borate anions. This process therefore comes at the expense of the corresponding capacities of the resin, which constitute key parameters in the production of ultrapure water. Laboratory measurements on SBA resins, some of which are doped with hydrogen carbonate, illustrate the severe extent of the effect described.

To prevent hydrogen carbonate formation, we at LANXESS package our ultrapure resins with highly regenerated anion exchange components in a special way to minimize CO<sub>2</sub> contamination during transportation and storage.

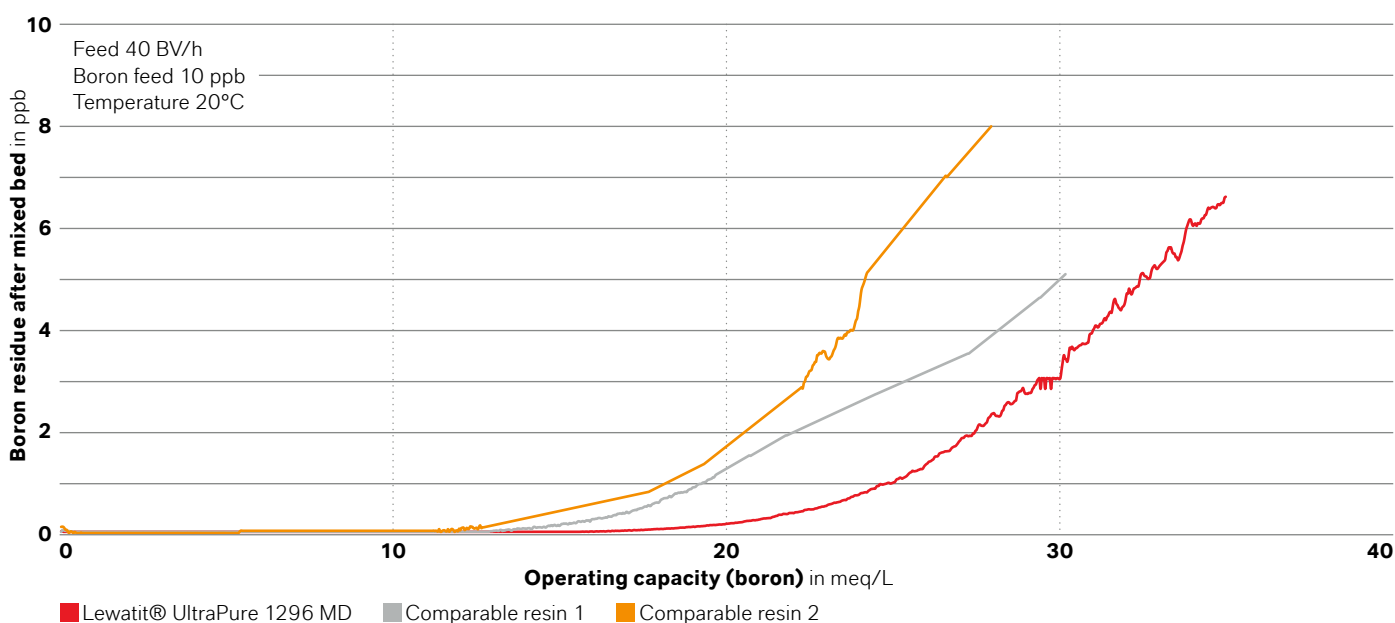
To do this, we use aluminum-coated liners in 200-liter drums and big bags, which form an efficient diffusion barrier for air and thus carbon dioxide.

Lewatit® UltraPure resins offer an additional benefit for borate removal in particular. A comparison of the borate capacity of Lewatit® UltraPure 1296 MD with two other common resins on the market reveals its superior retention capabilities even in the case of relatively long useful lives.



■ Boron removal control at LANXESS laboratory

**Figure 11: Lewatit® UltraPure 1296 MD contains of high capacity for boron**



# KEY CHARACTERISTICS OF LEWATIT® ULTRAPURE HIGH-END MIXED-BED SYSTEMS

## Lewatit® UltraPure 1292 MD

- Primary application: working mixed bed
- Clear visually identifiable separation of SAC and SBA due to similar bead size (mean bead size: SAC 0.65 / SBA 0.67 mm +/- 0.05 mm) and different bead colors
- Optimized, minimal release of organic substances after even short start-up phases
- High operating capacity even for weakly basic anions
- High mechanical and osmotic stability of the resin beads even after many regeneration cycles

## Lewatit® UltraPure 1294 MD

- Primary application: final polishing
- Highly regenerated resin components as delivered
- Optimized, minimal release of organic substances and particles after even short start-up phases
- Clear visually identifiable separation of SAC and SBA due to similar bead size and different bead colors
- High usable capacity even for weakly basic anions

## Lewatit® UltraPure 1296 MD

- Primary application: final polishing
- Optimized for maximum water quality for the semiconductor industry
- Continuous monitoring of resin production with regard to minimal release of ions, particles, and organic compounds
- Inhibited separation of resin components due to differences in bead size between SAC and SBA (mean bead size: SAC 0.55 mm / SBA 0.67 mm +/- 0.05 mm), meaning better exchange kinetics at high metric and specific flow rates.



■ Selection of Lewatit® UltraPure resin types

# AN OVERVIEW OF THE LEWATIT® ULTRAPURE RESIN RANGE



Strongly acidic cation exchange resins (SAC)	Lewatit® UltraPure 1221 MD	Lewatit® UltraPure 1213 MD	Lewatit® UltraPure 1216 MD	
Matrix	Styrenic	Styrenic	Styrenic	
Structure	Macroporous	Gel	Gel	
Functional group	Sulfonic	Sulfonic	Sulfonic	
Ionic form	Na <sup>+</sup>	H <sup>+</sup>	H <sup>+</sup>	
Total capacity, eq/L, min.	1.7	2.1	2.1	
Mean bead size, mm (+/- 0.05)	0.65	0.60	0.55	
Uniformity coefficient, max.	1.1	1.1	1.1	
Resistivity, MΩ·cm, min.	4**	12*	12*	
ΔTOC, ppb, max.	50**	10*	10*	

Weakly basic anion exchange resins (WBA)/ Strongly basic anion exchange resins (SBA)	Lewatit® UltraPure 1231 MD	Lewatit® UltraPure 1241 MD	Lewatit® UltraPure 1261 MD	Lewatit® UltraPure 1243 MD
Matrix	Styrenic	Styrenic	Styrenic	Styrenic
Structure	Macroporous	Gel	Macroporous	Gel
Functional group	3ry/4ry amine	4ry amine	4ry amine	4ry amine
Ionic form	Free base/Cl <sup>-</sup>	Cl <sup>-</sup>	Cl <sup>-</sup>	OH <sup>-</sup>
Total capacity, eq/L, min.	1.4	1.3	1.1	1.1
Mean bead size, mm (+/- 0.05)	0.59	0.62	0.65	0.67
Uniformity coefficient, max.	1.1	1.1	1.1	1.1
Resistivity, MΩ·cm, min.	4***	4***	4***	17*
ΔTOC, ppb, max.	50***	50***	50***	10*

SAC/SBA "Ready-to-use mixed beds"	Lewatit® UltraPure 1292 MD	Lewatit® UltraPure 1294 MD	Lewatit® UltraPure 1296 MD	Lewatit® UltraPure 1297 MD
Matrix	Styrenic	Styrenic	Styrenic	Styrenic
Structure	Gel	Gel	Gel	Gel
Functional group	Sulfonic /4ry amine	Sulfonic /4ry amine	Sulfonic /4ry amine	Sulfonic /4ry amine
Ionic form	H <sup>+</sup> /OH <sup>-</sup>	H <sup>+</sup> /OH <sup>-</sup>	H <sup>+</sup> /OH <sup>-</sup>	H <sup>+</sup> /OH <sup>-</sup>
Mean bead size, mm (+/- 0.05)	SAC 0.65 / SBA 0.67	SAC 0.65 / SBA 0.67	SAC 0.55 / SBA 0.67	SAC 0.35 / SBA 0.67
Resistivity, MΩ·cm, min.	18*	18*	18*	18*
ΔTOC, ppb, max.	10*	1.5*	1*	5*
Approx. ratio (cation:anion)	1:1 by eq	1:1 by eq	1:1 by eq	1:1 by eq

Specialties	Lewatit® K 7333
Matrix	Styrenic
Structure	Gel (Pd doped)
Functional group	4ry amine
Ionic form	OH <sup>-</sup>
Total capacity, eq/L, min.	1.1
Mean bead size, mm (+/- 0.05)	0.67
Uniformity coefficient, max.	1.1
Resistivity, MΩ·cm, min.	17*
ΔTOC, ppb, max.	10*

\* Rinsing 4 h 20 BV/h feed water quality 18.2 MΩ·cm 3 ppb TOC

\*\* After 200 g HCl reg. rinsing 4 h 20 BV/h feed water quality 18.2 MΩ·cm 3 ppb TOC

\*\*\* After 200 g NaOH reg. rinsing 4 h 20 BV/h feed water quality 18.2 MΩ·cm 3 ppb TOC



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#### **Health and safety information**

Appropriate literature has been assembled which provides information concerning the health and safety precautions that must be observed when handling the LANXESS products mentioned in this publication. For materials mentioned which are not LANXESS products, appropriate industrial hygiene and other safety precautions recommended by their manufacturers should be followed. Before working with any of these products, you must read and become familiar with the available information on their hazards, proper use and handling. This cannot be overemphasized. Information is available in several forms, e.g., material safety data sheets, product information and product labels. Consult your LANXESS representative in Germany or contact the Health, Safety, Environment and Quality Department (HSEQ) of LANXESS Germany or - for business in the USA - the LANXESS Product Safety and Regulatory Affairs Department in Pittsburgh, PA.

#### **Regulatory Compliance Information:**

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