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Guidelines for softening and desalination of industrial wastewater for reuse

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 282, *Water reuse*, Subcommittee SC 4, *Industrial water reuse*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

With the development of society and economy, the contradiction between water shortage and industrial growth is becoming increasingly acute. Industrial wastewater reclamation and reuse could be an effective way to alleviate this contradiction by improving the water utilization efficiency[4]. Industrial processes such as oil extraction, chemicals production, printing and dyeing, pharmaceuticals manufacturing and food processing[5] produce the wastewater containing total dissolved solids. In order to reuse these wastewater, total dissolved solids need to be removed by using water softening and desalination technologies[6].

Currently, wastewater softening and desalination processes are based on chemical precipitation, ion exchange, nanofiltration (NF), evaporation, reverse osmosis (RO), electrodeionization (EDI), electrodialysis (ED), membrane distillation (MD), and so on, see References [7] to [10]. Each technology has different applicable conditions and operational costs. The absence of an international standard to provide guidance on the selection of wastewater softening and desalination processes makes it difficult to determine the most appropriate softening or desalination technology for industrial enterprises. Therefore, it hinders industrial wastewater reclamation and reuse. Six technologies have been selected for consideration under this document, including chemical precipitation, ion exchange, nanofiltration (NF), reverse osmosis (RO), electrodialysis (ED), electrodeionization (EDI), and there are other technologies that could be similarly considered for future updates. It should be noted that mechanical vapour recompression (MVR) and multi-effect evaporation (MEE) are mainly used for evaporation and crystallization to acquire salts, not for the purpose of water reuse.

Based on the specific inorganic ion species and their concentration in influent, appropriate effluent quality can be obtained using the recommended technologies that meets the requirement for hardness, alkalinity and salinity for potential reuse applications.

This document is an innovative standard in the field of industrial wastewater reclamation and reuse. It can help enterprises, engineers, operators and other stakeholders, who engage in designing or operating in industrial saline wastewater reclamation and reuse, choose the technologies applying in the process, and evaluate the treatment effects. As a result, the reuse of industrial saline wastewater can be promoted and utilization of water can be improved.

Guidelines for softening and desalination of industrial wastewater for reuse

1 Scope

This document provides guidance on, the evaluation and comparison of wastewater softening and desalination processes for industrial wastewater reclamation and reuse with specific consideration for the following six: 1) chemical precipitation; 2) ion exchange; 3) nanofiltration (NF); 4) reverse osmosis (RO); 5) electrodialysis (ED) and 6) electrodeionization (EDI). This document provides guidance on the characterisation of both influent and effluent quality (e.g. hardness, alkalinity, etc.) and the effects of these processes on those constituents. The purpose of softening and desalination is only for the reuse usages that have requirements for hardness and salinity, such as cooling circulating water, boiler water, production process water, and cleaning water.

This document includes the following sub-processes of wastewater softening and desalination processes:

a) wastewater softening processes based on chemical precipitation, ion exchange and NF, which aim to remove hardness ions, such as Mg^{2+} and Ca^{2+};

b) desalination processes based on ion exchange, RO, ED, EDI and NF, which aim to remove the most of total dissolved solids (TDS).

This document is applicable to:

a) industrial saline wastewater, which has been pre-treated to remove most of the organic matters if necessary;

b) the selection or design of wastewater softening and desalination processes for reuse of wastewater from industries.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 20670, *Water reuse — Vocabulary*

3 Terms, definitions and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO 20670 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1 Terms and definitions

3.1.1
regeneration
process of restoring an ion-exchange resin after use to its operationally effective state

Note 1 to entry: Two types of generation can occur: co-current regeneration and counter-current regeneration. Co-current regeneration is original downflow process where both input water and regeneration chemicals flow in the same direction, while counter-current regeneration is upflow process where input water and regeneration chemicals flow in different directions.

3.1.2
electrodeionization
method for removing ions by combination of mixed bed ion exchange and electrodialysis in an electrodialyser, where the fresh water chamber is filled with mixed bed ion exchange resin, and the ion exchange resin can be electrochemically regenerated by polarization during the electrodialysis process

Note 1 to entry: Generally, it is a polishing process for production of ultrapure reclaimed water and used after reverse osmosis.

3.1.3
electrodialysis
process used for the deionization of water in which ions are removed, under the influence of an electric field, from one body of water and transferred to another across an ion-exchange membrane

[SOURCE: ISO 6107-1:2004, 32]

3.1.4
industrial saline wastewater
industrial wastewater that contains high concentration of inorganic ions

3.1.5
ion exchange
process by which certain anions or cations in water are replaced by other ions by passage through a bed of ion-exchange material

[SOURCE: ISO 6107-1:2004, 46]

3.1.6
mechanical vapour recompression
use of the heat of the secondary steam as a heat source instead of fresh steam by raising its temperature, with a part of the compressor working to achieve cyclic evaporation

3.1.7
membrane distillation
separation process where a micro-porous hydrophobic membrane separates two aqueous solutions at different temperatures

3.1.8
microfiltration
type of physical filtration process by pressure driven where a contaminated liquid is passed through a special pore-sized membrane (0,1-1 µm) to separate microorganisms and suspended particles from process liquid

3.1.9
multi-effect evaporation
use of microporous membranes with a filtration accuracy of 0,01-0,1 µm for the separation of microorganisms, large molecules or very finely divided suspended matter from water by filtration, often by means of applied differential pressure

3.1.10
nanofiltration
membrane separation technology with a filtration accuracy of 0,001-0,01 µm to separate proteins and low molecular organic compounds

3.1.11
precipitation
chemical reaction in solution resulting in the formation of a solid product

[SOURCE: ISO 11074:2015, 6.4.30]

3.1.12
pre-treatment
treatment process or processes carried out before the softening and desalination processes

3.1.13
reverse osmosis
flow of water through a membrane with a filtration accuracy of 0,000 1-0,001 µm, from a more concentrated to a less concentrated solution, as a result of applying pressure to the more concentrated solution in excess of the normal osmotic pressure

Note 1 to entry: The filtration accuracy of membrane is added.

[SOURCE: ISO 6107-1:2004, 61]

3.1.14
softening
partial or complete removal from water of calcium and magnesium ions which are responsible for hardness

Note 1 to entry: In this context, not only calcium and magnesium ions are removed, other inorganic ions and cations are also included.

[SOURCE: ISO 6107-1:2004, 68]

3.1.15
ultrafiltration
use of microporous membranes with a filtration accuracy of 0,01-0,1 µm for the separation of large molecules or very finely divided suspended matter from water by filtration, often by means of applied differential pressure

Note 1 to entry: The filtration accuracy of microporous membranes is added.

[SOURCE: ISO 6107-6:2004, 100]

3.2 Abbreviated terms

BOD_5 biochemical oxygen demand after 5 days

COD chemical oxygen demand

DO dissolved oxygen

ED electrodialysis

EDI electrodeionization

MF microfiltration

NF nanofiltration

RO	reverse osmosis
TDS	total dissolved solids
TOC	total organic carbon
TSS	total suspended solids
UF	ultrafiltration
MD	membrane distillation
MEE	multi-effect evaporation
MVR	mechanical vapor recompression

4 General

Water quality indicators should include TSS, TOC, COD, pH, temperature, TDS, the species and concentrations of ions.

The product water from wastewater softening and desalination processes is recommended to be reused for urban non-potable water, environmental water, and as pure or ultrapure water for cooling water, boiler feed water, process water, rinse water, and so on[4].

The process selection of wastewater softening and desalination processes should be determined after technical and economic comparison based on factors such as influent quality, product quality, quantity requirements, site conditions and environmental protection requirements.

The wastewater needs to be pre-treated if necessary, before being fed into softening and desalination devices.

The selection of pre-treatment process should consider the quality of wastewater, influent quality requirements for softening and desalination processes, water treatment volume and test data. Besides, the operational experience of similar projects should be referred, combined with local conditions. Finally, users can determine which technology to adopt through technical and economic comparison.

Minimizing the discharge quantity of waste acid, waste alkali, waste residue and other harmful substances are important in the selection of softening and desalination processes or device. Measures for treating and disposing these wastes should be taken to meet the relevant environmental protection requirements.

Waste liquid (e.g., regeneration liquid of ion exchange resin process, concentrate of reverse osmosis process, etc.) disposed from the softening and desalination processes should be collected separately according to the characteristics of wastewater quality.

Process flow diagram of industrial saline wastewater treatment for reuse is shown in Figure 1.

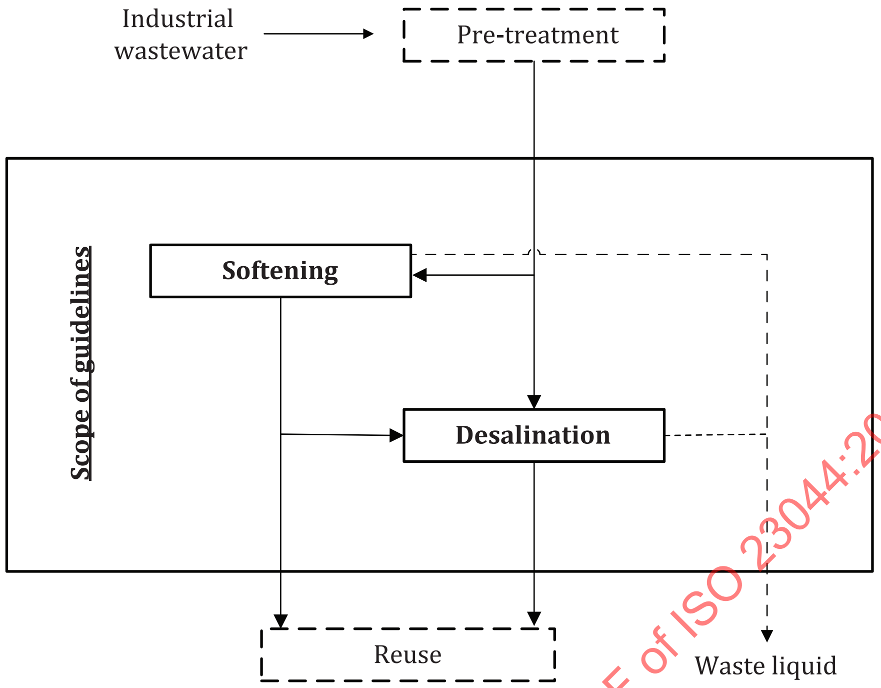

Figure 1 — Process flow diagram of industrial saline wastewater treatment for reuse

5 Requirements for influent quality

Influent quality requirements for softening and desalination device are shown in Table 1. It is noted that data provided in this table is all in advisory typical ranges, which is suggested to be applied according to specific conditions, as well as the manufacturer's specifications. The parameters listed in Table 1 are also illustrated as follows to show its effect on softening and desalination devices.

a) Silt density index (SDI) reflects the content of particles, colloids, and other objects in influent that can block softening and desalination devices. SDI values higher than the limit can easily block the membrane which will lead to fouling, thereby shortening the operating life of the membrane.

b) Turbidity represents the concentration of undissolved matters in influent that reduce transparency. These undissolved matters can adhere to surface of ion exchange resin, and then block the exchange channel or pollute resin. It can also cause membrane fouling.

c) Water temperature can affect ion exchange rate and ion absorption ability of resin. It also can affect membrane flux and TDS removal ability of membrane.

d) pH can affect TDS removal ability of membrane and shorten its operating life if exceed typical range.

e) Chemical oxygen demand refers to organic matters which can easily pollute anion exchange resin, because it is difficult to precipitate after the reaction with the anion exchange resin.

f) Appropriate residual chlorine can ensure the sterilization ability for water quality. However, resin is combined with macromolecular organic compounds those can be easily oxidized by high concentration of chloride to break the chemical structure, and then ion exchange ability of resin would be weakened. High residual chlorine can also oxide membrane element and make an irreparable damage[11].

g) Iron and manganese can be intercepted by resin to form adsorbent that is not easy to wash off. The resin would lose function as the reaction is not reversible. In addition, both iron and manganese can accelerate the oxidation of the membrane and cause irreversible damage to the membrane element.

h) Lower electrical conductivity reflects lower ion content, which is beneficial to form a larger electric potential gradient. More cations and anions would be generated along with the increasing of water dissociation degree. Then the regeneration ability of resin can keep well.

i) High total exchangeable anions can reduce the resistivity of effluent, and then larger running current should be set. However, larger running current can increase the system current and residual chlorine, which is not beneficial to the membrane.

j) High hardness (>200-500 mg/l $CaCO_3$) can cause fouling in EDI units. Low hardness (<200 mg/l $CaCO_3$) can extend the cleaning cycle and improve the water utilization rate of EDI system.

k) Both CO_2 and SiO_2 are weakly ionized substances, which can lead to deterioration of water quality. Decarbonator is suggested to be installed to remove CO_2.

Table 1 — Influent quality requirements for softening and desalination devices

Parameter		Ion exchange	Nanofiltration or reverse osmosis	Electrodialysis	Electro-deionization
Silt Density Index (SDI_{15})		<5	<5[a]	<5	<1
Turbidity (FTU)[b]	Counter-current regeneration	<2	<1	<1	<1
	Co-current regeneration	<5			
Water temperature (°C)		5~40[c]	5~35[d]	5~40	5~40
pH (25 °C)		—[e]	3~11	4~10	5~9
Chemical oxygen demand (mg/l) ($K_2Cr_2O_7$)		<50	—	<10	—
Residual chlorine (mg/l)		<0,1	<0,1, control to 0	<0,2	<0,05
Fe (mg/l)		<2[f]	<0,05 (DO > 5 mg/l)[g]	<0,3	<0,01
Mn (mg/l)		—	<0,3	<0,1	<0,01
Electrical conductivity (25 °C, µS/cm)		—	—	>10 000[h]	<40[i]

[a] RO membrane manufacturers recommend that the RO feed water should have an SDI < 5. However, through long term operational experience, many operators now recommend having an SDI < 3[12].

[b] FTU: formazan turbidity units.

[c] Higher water temperature can increase ion exchange rate, but if water temperature is too high, the ion absorption ability will weaken. Besides, resin may deteriorate and radical group for exchange may degrade with too high temperature. The influent temperature for alkali II resin and acrylic resin is suggested to be lower than 35 °C.

[d] Higher water temperature can increase membrane flux, but TDS removal ability will be worse if water temperature is too high. Optimum water temperature for reverse osmosis devices is suggested to be in the range of 20~25 °C.

[e] The symbol "—" means that any value for the given parameter can be applicable.

[f] The influent iron concentration of ion exchange resin device, which is regenerated by hydrochloric acid and sulfuric acid, is suggested to be less than 2 mg/l, and the amount of iron contained in the influent of sodium ion exchange resin device for softening is suggested to be less than 0,3 mg/l.

[g] The oxidation rate of iron depends on the iron content, the concentration of DO in water and the pH value of water[13]. When the pH is less than 6, DO is suggested to be less than 0,5 m g/l, and maximum Fe^{2+} is suggested to be less than 4 mg/l.

[h] It is not economical to utilize electrodialysis units if the conductivity of the influent is less than 10 000 µS/cm.

[i] The influent into electrodeionization device is suggested to be the effluent from reverse osmosis device, whose expected value of conductivity (25 °C), including the equivalent conductivity of carbon dioxide, should be less than 20 µS/cm.

[j] When the hardness is greater than 500 mg/l ($CaCO_3$), the exchange capacity of ionic resin may reach saturation fast.

[k] When the hardness is greater than 200 mg/l ($CaCO_3$), scaling may generate in reverse osmosis membrane. When the hardness is greater than 500 mg/l ($CaCO_3$), scaling may generate in nanofiltration membrane.

Table 1 *(continued)*

Parameter	Ion exchange	Nanofiltration or reverse osmosis	Electrodialysis	Electro-deionization
Total exchangeable anions (mg/l, $CaCO_3$)	—	—	—	<25
Hardness (mg/l, $CaCO_3$)	<500[j]	<200 (RO) <500 (NF)[k]	<10	<1
CO_2 (mg/l)	—	—	—	<5
SiO_2 (mg/l)	—	—	<20	≤0,5

[a] RO membrane manufacturers recommend that the RO feed water should have an SDI < 5. However, through long term operational experience, many operators now recommend having an SDI < 3[12].

[b] FTU: formazan turbidity units.

[c] Higher water temperature can increase ion exchange rate, but if water temperature is too high, the ion absorption ability will weaken. Besides, resin may deteriorate and radical group for exchange may degrade with too high temperature. The influent temperature for alkali II resin and acrylic resin is suggested to be lower than 35 °C.

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[j] When the hardness is greater than 500 mg/l ($CaCO_3$), the exchange capacity of ionic resin may reach saturation fast.

[k] When the hardness is greater than 200 mg/l ($CaCO_3$), scaling may generate in reverse osmosis membrane. When the hardness is greater than 500 mg/l ($CaCO_3$), scaling may generate in nanofiltration membrane.

Industrial wastewater should be pre-treated before being fed into the softening and desalination devices to improve water quality by removing particles, TSS, organic matters, etc. Pre-treatment processes consist of conventional treatment and tertiary treatment. Popular pre-treatment processes are shown in Annex A. Combination of those technologies can be adopted according to the quality of wastewater, influent quality requirements of softening and desalination processes, technical feature, cost and so on. Besides, experimental data from lab scale study or similar engineering experience also should be referred.

6 Softening process

Based on hardness and alkalinity of the influent and the water quality requirements of the effluent, softening process can adopt chemical precipitation, ion exchange resin or a combination of the two technologies[9]. Recommended softening processes are illustrated as follows. Table 2 can also be referred for softening process selection[6][10].

a) The effects of ion exchange methods are stable and accurate, including single-stage sodium resin, two-stage sodium resin, H-type resin in series with sodium resin, and weak acid cation resin, etc.

b) As to chemical precipitation, the most common precipitants are lime, soda ash and sodium hydroxide.

c) Single-stage sodium resin cannot remove alkalinity; thus, it is suitable for treatment of wastewater with low alkalinity and hardness.

d) Neither can two-stage sodium resin remove alkalinity. The two-stage sodium resin is applicable to treat wastewater with low alkalinity but high hardness.

e) Lime-soda ash softening is an option. Lime softening can remove carbonate alkalinity and soda ash can remove non-carbonate alkalinity.

f) Lime softening combined sodium resin can simultaneously remove hardness and alkalinity, so it is suitable for wastewater with high hardness of carbonate, low excess alkalinity or no excess alkalinity. Lime softening treatment has the advantage of low cost, but there are shortcomings like labour intensity, poor working conditions and that non-carbonate calcium hardness is not affected by treatment with lime softening alone.

g) H-type resin in series with sodium resin is suitable for wastewater with high hardness and high alkalinity.

h) H-type resin in parallel with sodium resin can simultaneously remove hardness and alkalinity, so it is suitable for wastewater with high hardness of carbonate and high alkalinity.

i) Weak acid cation resin in hydrogen form can remove alkalinity but not suitable for the removal of hardness.

j) Nanofiltration membranes can intercept calcium and magnesium ions in water, radically reducing the hardness. However, it has strict requirements for the pressure of influent and high investment and operation cost.

Table 2 — Softening process selection

Number	Process name and code	Influent quality			Effluent quality	
		Total hardness [mg/l ($CaCO_3$)]	Carbonate hardness [mg/l ($CaCO_3$)]	Ratio of carbonate hardness to total hardness	Hardness [mg/l ($CaCO_3$)]	Alkalinity [mg/l ($CaCO_3$)]
1	Ion exchange[a]	<500	<350	—	<20	5
2	Chemical precipitation[b]	—	—	—	≥35	≥135
3	lime softening in series with sodium resin (CaO-Na)	—	>150	>0,5	<2	40~60
4	Nanofiltration	<500	<350	—	<20	0

[a] Table symbol: Na – Sodium ion exchanger; CaO – Lime softening treatment device; "—" means that any value for the given parameter can be applicable.

Weak acid cation exchangers in hydrogen form are used alone to remove carbonate hardness.

The effluent hardness of the weak acid cation exchanger in hydrogen form is equal to the sum of the non-carbonate hardness of the raw water and the effluent alkalinity. Effluent alkalinity refers to average effluent alkalinity.

Sodium-based resins use sodium ions as exchange ions to exchange calcium and magnesium in water, while hydrogen resins use hydrogen ions as exchange ions to exchange calcium and magnesium.

The higher the total hardness is, the easier it is for the resin to reach saturation, shorten the cleaning period and reduce the service life of the resin.

[b] When lime is used for softening, the raw water should be heated to 30~40 °C and ferric salt should be used as coagulant.

Lime addition is suitable for water with low non-carbonate hardness and high carbonate hardness, while lime and soda ash are suitable for water with high hardness of non-carbonate.

Sodium hydroxide reveals better softening performance than lime, but its cost is relatively high, which is applicable for circumstances with high requirements on hardness remove.

7 Desalination process

Desalination processes based on ion exchange resin, RO, ED, EDI, and nanofiltration are suggested to be adopted according to influent and effluent quality requirements. The suggestion of desalination processes can be found in Table 3[4][6]. Other desalination technologies and their performance are shown in Annex B.

As to ion exchange technology, if the ratio of strong acid anion to weak acid anion in the influent is relatively stable, in the primary desalination process, unit series system of cation and anion exchangers can be adopted, and resin volume of the anion exchanger should be the calculated value with 10 %~15 % margin plus.

If the ratio of strong acid anion to weak acid anion in the influent is changeable, in the primary desalination process, the parallel system of master control of the cation and anion resin exchanger should be adopted.

Electrodialysis is often combined with reverse osmosis. When the reverse osmosis process is placed behind, the reverse osmosis feed water is the fresh water of electrodialysis. When the reverse osmosis equipment is placed in front, the electrodialysis feed water is the concentrated water of reverse osmosis.

The influent of EDI should be the effluent from RO with low hardness and conductivity. The effluent from EDI can be used as ultrapure water in industrial production.

For ion exchange technology, both cation exchange on the hydrogen cycle and anion exchange on the hydroxyl ion cycle are required to achieve a reduction in dissolved solids.

Cation exchange resin can be regenerated with acid (e.g., sulfuric acid), while anion exchange resin can be regenerated with alkali (e.g., sodium hydroxide). For anion exchangers, alkali regeneration solution should be heated when silicon content in influent is relatively high.

Table 3 — Typical desalination process selection

Number	Process name and code	Influent quality				Effluent quality	
		Alkalinity [mg/l ($CaCO_3$)]	Strong acid anions [mg/l ($CaCO_3$)]	Total dissolved solids [mg/l]	SiO_2 [mg/l]	Conductivity [25°C, µS/cm]	SiO_2 [µg/l]
1	Ion exchange (single bed)[a]	—[b]	—	<500	—	<10	<100
2	Ion exchange (single bed) → Ion exchange (mixed bed)[c]	—	—	<500	—	<0,2	<100
3	One-stage reverse osmosis RO[d]	—	—	500~50 000	—	<15	<500
4	Two-stage reverse osmosis RO→RO	—	—	500~50 000	—	<10	<500
5	Nanofiltration NF	—	—	300~ 30 000[e]	—	<0,1	—
6	Electrodialysis ED	—	—	10 000~ 200 000	<20	<1 000	<10 000
7	Electrodialysis→ Ion exchange	—	—	10 000~ 200 000	<20	<10	<100
8	Electrodialysis→ Reverse osmosis ED→RO	—	—	10 000~ 200 000	<20	<15	<500
9	Reverse osmosis→ Electrodialysis RO→ED	—	—	500~50 000	<20	<1 000	<500

[a, c] All kinds of ion exchange resins have certain heat resistance properties. Hence, there should be strict control over water temperature when used.

When the alkalinity is less than 200 mg/l ($CaCO_3$), the first order of desalination can be used. If it is over 200 mg/l ($CaCO_3$), the second order desalination can be used.

When the carbonate hardness is greater than 150 mg/l ($CaCO_3$), these desalination processes based on ion exchange can be used, such as the first order desalination of weak acid, the first order desalination of weak acid and mixed bed, the first order desalination of weak acid and alkali, depending on different situations.

Ion exchange resins are of various types. The specific implementation needs further consideration to select the most applicable product.

[b] Symbol in table: "—" means that any value for the given parameter can be applicable.

[c] When the quality of the effluent is not so strict, the conductivity of the mixed-bed resin can be controlled to be less than 0,20 µS/cm.

[d] RO membrane can be classified in many types, according to different salinity, materials, etc. In this standard, RO is a general term, not a specific implementation or product. The actual application should be selected based on the specific situation.

[e] The requirements of TDS influent for nanofiltration is closely related to ion composition. High TDS influent can meet the requirements when the content of one-valent ions in raw water is high.

Table 3 *(continued)*

Number	Process name and code	Influent quality				Effluent quality	
		Alkalinity [mg/l ($CaCO_3$)]	Strong acid anions [mg/l ($CaCO_3$)]	Total dissolved solids [mg/l]	SiO_2 [mg/l]	Conductivity [25°C, µS/cm]	SiO_2 [µg/l]
10	Reverse osmosis →Ion exchange (single bed) →Ion exchange (mixed bed)	—	—	500~50 000	—	<0,1	<10
11	Two-stage reverse osmosis→ Electrodeionization RO→RO→EDI	—	—	500~50 000	—	<0,10	<10

[a, c] All kinds of ion exchange resins have certain heat resistance properties. Hence, there should be strict control over water temperature when used.

When the alkalinity is less than 200 mg/l ($CaCO_3$), the first order of desalination can be used. If it is over 200 mg/l ($CaCO_3$), the second order desalination can be used.

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