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Conventional Resin Cation Exchangers versus EDI for CACE Measurements – An Ion Chromatography Study

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ABSTRACT

As a result of the simple measuring principle, the sensitivity and its high reliability, the online measurement of conductivity after water has passed through a column of strongly acidic cation exchange resin has become one of the most commonly used analytical method in power plants with steam generators.

However, the limited amount of cation exchange resin in the exchanger column is spent after some time and the alkalizing agent breaks through, resulting in false high conductivity measurements. Therefore, the cation exchange resin has to be replaced on a regular basis, which leads to an interruption of the measurement. In power plants with a high pH in the feedwater, such as in the secondary circuit of pressurized water reactors or in frequently cycling power plants, the resin has to be changed frequently. At high pH, the problems with the discontinuous measurement of the conductivity after a cation exchange resin, such as the high maintenance effort and resin consumption, become obvious. Here, cation exchange with an electro-deionization (EDI) device might offer a continuous alternative.

This paper provides a comparison to the classic design using a passive cation exchange resin, showing that constantly lower CACE values can be achieved with the EDI device. The effects of the ion exchange resin and the EDI have been analyzed using ion chromatography, providing a detailed chemical picture of the relevant processes influencing the measurement results.

INTRODUCTION

In order to minimize corrosion in a power plant, the water-steam cycle chemistry must be monitored at all times. Therefore, a continuous and thorough measurement of the key parameters is necessary. Especially the ingress of corrosive anions, such as chloride and sulfate, should be monitored permanently.

The conductivity measurement after a cation exchanger (CACE) in power plants with steam turbines was introduced soon after 1950 [1]. The measurement has been proven to be a simple and reliable tool to detect anionic impurities in the water steam cycle. Though it is an empiric sum parameter, measuring all anions together and not specific concentrations, it turns out to be reactive to very small ion concentrations, making it one of the most important measurements for monitoring the quality of water and steam in a thermal power plant [2].

In this paper, we compare two techniques to achieve the cation exchange prior to the conductivity sensor and their impact on the CACE measurement. First, there will be a short chemical recapitulation of CACE, and then the conventional cation exchange and cation exchange using electro-deionization (EDI) will be explained. The CACE values obtained with both techniques in the laboratory will be compared. If indeed there is an influence of the measurement on the CACE, the results are not necessarily expected to be identical. Every ion liberated by the monitor will result in either too high (leakage of anions) or too low (leakage of cations) CACE readings. A simple laboratory experiment was designed showing the typical behavior of an EDI module compared to a conventional cation exchange column. Deviations observed between the two techniques will be explained using ion chromatography.

CHEMICAL BACKGROUND OF CACE

In principle, the presence of ions in pure water could be easily detected by directly measuring its specific conductivity. Adding 40 $\mu g \cdot kg^{-1}$ of sodium chloride to pure water will roughly triple its conductivity from 0.055 $\mu S \cdot cm^{-1}$ to 0.140 $\mu S \cdot cm^{-1}$ (Table 1).

This difference can easily be detected with standard conductivity electrodes. However, the water in the water-steam cycle of power plants is alkalized to pH 9 to pH 10 in order to minimize corrosion. Usually ammonia or small amines are added to the water to increase the pH to the

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	Contaminant	Specific Conductivity [µS · cm^{-1}]
Ultra-pure water	—	0.055
	40 µg · kg^{-1} NaCl	0.140
Water alkalized with NH_3 to pH 10	—	27.15
	40 µg · kg^{-1} NaCl	27.23

Table 1:
Specific conductivities of ultra-pure water and alkalized water, before and after the addition of 40 µg · kg^{-1} of sodium chloride.

	Contaminant	CACE [µS · cm^{-1}]
Water alkalized with NH_3 to pH 10	—	0.055
	40 µg · kg^{-1} NaCl	0.305

Table 2:
CACE of alkalized water, before and after the addition of 40 µg · kg^{-1} of sodium chloride.

desired value. However, the conductivity of the water is then no longer governed by the presence of ionic impurities, but by the concentration of the alkalizing agent.

In the example above, where the pH is adjusted to pH 10 by the addition of ammonia, the detection of 40 µg · kg^{-1} of sodium chloride would mean discriminating between 27.15 µS · cm^{-1} and 27.23 µS · cm^{-1} (Table 1), which is hard to achieve reliably. Therefore, the specific conductivity cannot directly be used to monitor the ingress of corrosive anions.

In order to reduce the conductivity coming from the alkalizing agent, it is replaced by protons in a cation exchange step before the conductivity is measured. The alkalizing agent is removed from the sample, yielding water in the net reaction (Eq. (1)), while neutral salts are transformed to the corresponding acids (Eq. (2)):

$$NH_4^+ + OH^- \rightarrow H^+ + OH^- \rightarrow H_2O \quad (1)$$

$$Na^+ + Cl^- \rightarrow H^+ + Cl^- \quad (2)$$

The corresponding conductivity is called conductivity after cation exchange (CACE) or acid conductivity. The CACE measurement for the abovementioned example, water alkalized to pH 10 with ammonia, then reads as in Table 2.

Here, the conductivity even increases by more than 0.2 µS · cm^{-1}, as hydrochloric acid is measured and not sodium chloride as in the first example. The key step to a robust CACE measurement is therefore the removal of the alkalizing agent and other cations before the measurement without changing the rest of the sample.

CACE MEASUREMENTS USING CATION EXCHANGE COLUMNS

Conventionally, the alkalizing agent is removed by passing the sample through a small cation exchange column prior to the conductivity sensor. The alkalizing agent is literally trapped in the resin and protons are liberated from the resin. This technique has been proven to be simple and robust, with a high degree of cation retention.

In practice, it has been noticed that the exact setup of the measurement has an influence on the baseline CACE that can be achieved. The installation itself, in particular the diameter and length of the column and the quality of the resin chosen, but also operational parameters, such as sample flow and temperature or fresh versus regenerated resin, affect the CACE baseline. It was already noticed by Sisson and colleagues that there is a contribution to the CACE coming from the resin. They attributed this to sulfate ions leaking from the resin [3]. The effective contact time between the sample and the resin thus has an impact on the CACE reading. The use of regenerated resin instead of fresh resin on the other hand bears the risk of ammonia leakage through the column leading to too low CACE readings.

However, the biggest practical problem with the current CACE monitors is the consumption of the resin during the measurement. The resin is exhausted after a given amount of alkalizing agent is passed through the column. Figure 1 shows the theoretical lifetime of one liter of cation exchange resin as a function of sample pH. The resin has to be replaced after this period at the latest to guarantee a valid measurement.

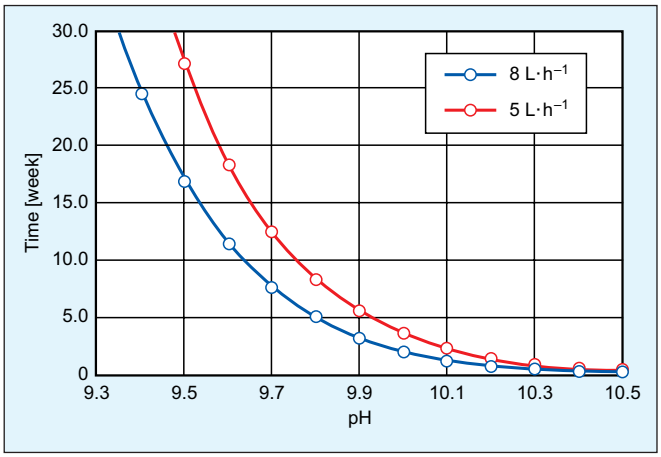

Figure 1:
Time until one liter of cation exchange resin is exhausted as a function of the sample pH. For the calculation a resin capacity of 2 mol · L^{-1} was assumed and ammonia was the only alkalizing agent. Values for two different sample flows (8 L · h^{-1} and 5 L · h^{-1}) are shown.

The frequent exchange of the resin not only means a significant workload for the plant operational personal, but there is also always a period with invalid CACE readings associated with the exchange of the cation exchange column and the rinse-down time of the resin. It would therefore be beneficial to replace the discontinuous cation exchange with a continuous removal of the alkalizing agent.

CACE MEASUREMENTS USING EDI

In recent years, water deionization using electro-deionization (EDI) has become more and more reliable and widespread in water desalination plants. The advantage of EDI is that the ion exchange resin is regenerated in place, leading to a continuous process. Ions present in the feedwater pass through ion exchange membranes to a concentrate stream and are thus separated from the product, called the dilute. The protons and hydroxide ions needed for regeneration are generated by water electrolysis, eliminating the use of regeneration chemicals. A schematic drawing of such an EDI device is shown in Figure 2.

The use of such a miniaturized EDI cell for the CACE measurement was proposed and characterized by Lok and colleagues [5] and Bengtsson and colleagues [6]. Instead of the whole EDI process, only the first step, where cations are removed, is carried out and the conductivity is recorded afterwards. Based on their promising results [5,6] SWAN designed an EDI module that is able to

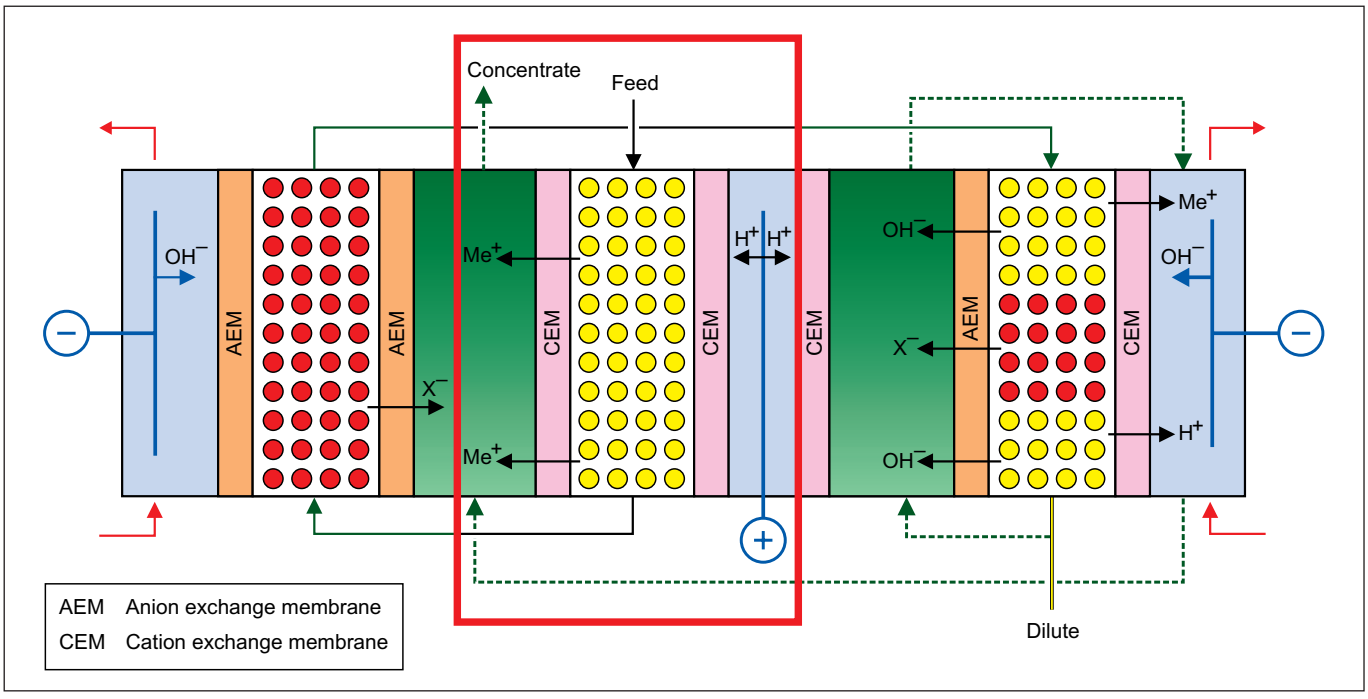

Figure 2:
EDI module for water deionization. The water is first passed over a cation exchange bed (shown in yellow) and then over an anion exchange bed (shown in red); in a polishing step it is passed over a mixed bed. Protons and hydroxide ions are constantly generated through water electrolysis at the electrodes and are used to regenerate the resins. Ion exchange membranes allow the ions present in the feedwater to pass from the sample chambers into the concentrate. Using the EDI technique for cation exchange prior to a conductivity measurement would only require the first exchange step shown in the center, highlighted in the red box. The sample is passed only over the cation exchange bed and the dilute is used for the CACE measurement. Figure adapted from reference [4].

eliminate the alkalizing agent up to specific conductivities over 40 $\mu S \cdot cm^{-1}$ in a single exchange step.

The performance of three of these prototypes has been compared to a standard CACE monitor (AMI Deltacon) in the laboratory. A setup was designed that allowed the parallel measurement of the four monitors while dosing ammonia as the alkalizing agent and sodium chloride as a model for ionic impurities. Figure 3 shows the baseline CACE obtained (on the left) and the response to sodium chloride (on the right).

The baseline CACE obtained with the EDI monitors is about 60 $nS \cdot cm^{-1}$ lower than with the cation exchange resin. In theory, the CACE should be 56 $nS \cdot cm^{-1}$ as this is the specific conductivity of the water used before ammonia dosing. There are two possible reasons for the higher CACE baseline. First, there is potentially some carbon dioxide dissolved in the ammonia stock solution. The resulting hydrogen carbonate ions contribute to the CACE reading. As the monitors measure in parallel, this contribution should be identical for all four of them. Second, there might be a contribution coming from the monitor itself, in particular the resin. Here again, there are two options, either leakage of anions leading to too high CACE values or leakage of cations leading to too low CACE values. The difference of 60 $nS \cdot cm^{-1}$ between the EDI technique and the cation exchange column is most likely caused by ion leakage.

The response to sodium chloride dosing is fast in all four cases, but the EDI readings show a slightly lower value than the cation exchange column. This observation is even true when a constant offset is added to the EDI readings (as is done in Figure 3 on the right), such that the baseline CACE is identical for all monitors. This effect again can be explained either by cation leakage or by chloride loss through the EDI process. Interestingly, the baseline CACE as well as the response to chloride ingress is very reproducible among the three EDI monitors.

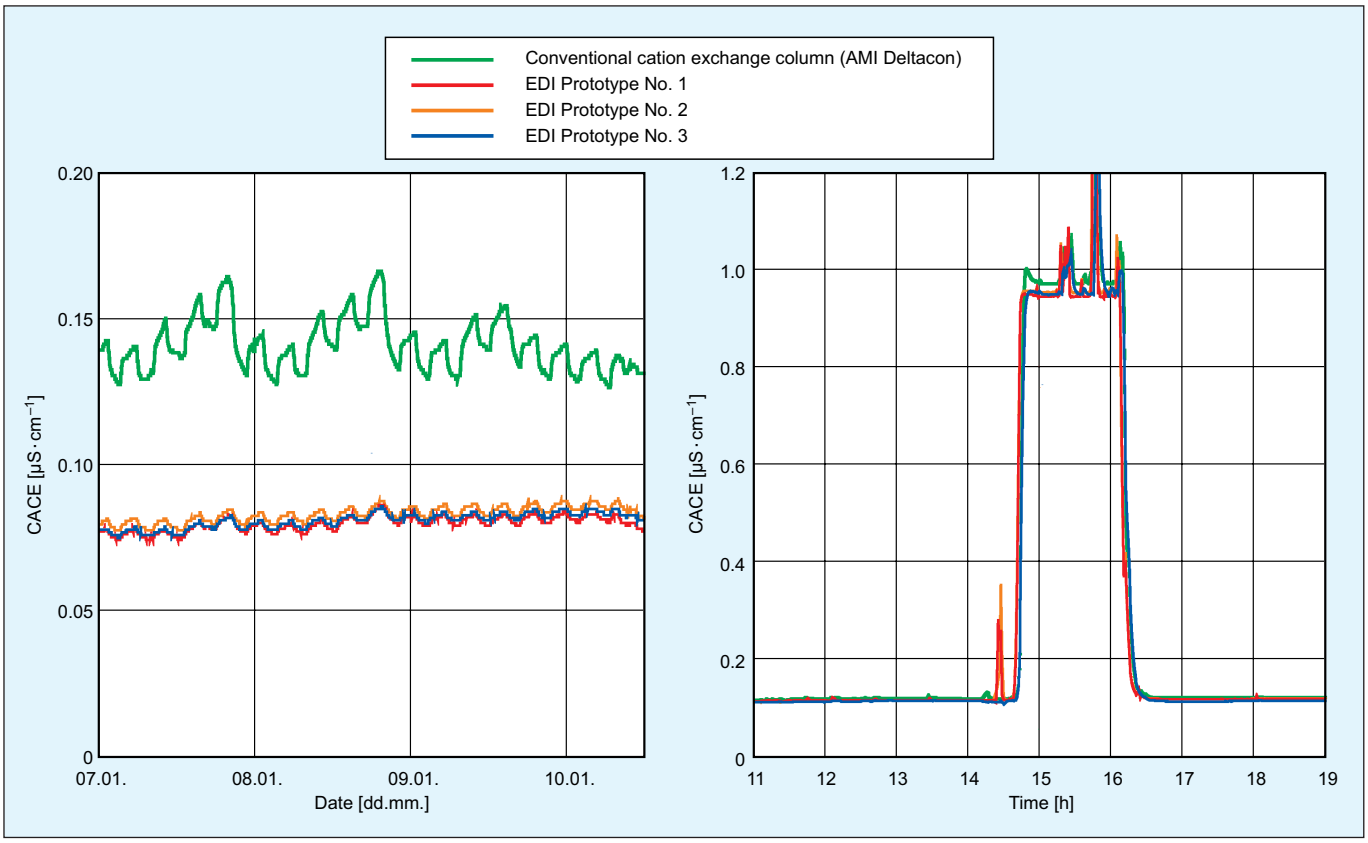

Figure 3:
Comparison of CACE readings measured in the laboratory in parallel with a conventional cation exchange column (AMI Deltacon, shown in green) and three prototypes using EDI (shown in red, orange and blue). The pH of the sample water was adjusted with ammonia to pH 9.8, corresponding to a specific conductivity of 17 $\mu S \cdot cm^{-1}$. On the left a period of four days with constant ammonia dosing is shown. It can be seen that the EDI modules give a lower CACE baseline, closer to the theoretical value of 55 $nS \cdot cm^{-1}$. The ripples are caused by temperature variations in the incoming water. On the right the response of the four monitors with respect to sodium chloride is shown. The dosing shown corresponds to roughly 70 $\mu g \cdot kg^{-1}$ chloride. An offset of 60 $nS \cdot cm^{-1}$ was added to the EDI measurements in order to have the same CACE baseline. The response of the EDI modules is however still a few percent lower than the response of the Deltacon. Spikes in the data correspond to instabilities in the flow in the dosing system and are correctly detected by all monitors.

ANALYSIS OF CACE RESULTS WITH ION CHROMATOGRAPHY

In order to test if the pronounced offset of the baseline CACE is indeed sulfate leakage from the resin as proposed by Sisson and colleagues [3] and if there is (additional) cation leakage through the EDI, the water at the inlet and the outlet of the monitors was analyzed using ion chromatography (IC). In addition, we wanted to see whether there is a chloride loss during the EDI process, which would explain the slightly lower response to the chloride dosing of the EDI monitors.

Due to the expected concentrations in the lower ppb range, samples for the IC were taken online directly from the effluent without any manual sampling or transfer into vials. In order to exclude contributions from impurities of the used ammonia and carbonate forming in the ammonia stock solution, we did not alkalize the sample for the IC measurement. So only highly purified water with a constant conductivity of 56 nS · cm^{-1} and a sodium chloride stock solution were used in the following experiment.

Figure 4 shows the CACE readings of all four monitors. Three different concentrations of chloride were dosed, corresponding approximately to 20 µg · kg^{-1}, 45 µg · kg^{-1} and 70 µg · kg^{-1} of chloride ions. All four monitors responded quickly to the chloride ingress, with slightly lower, but very reproducible, values of the EDI monitors. In Figure 4 there is already an offset added to the EDI signals to adjust the baseline CACE. The offset between both techniques increases with increasing chloride concentration. The EDI shown in orange had twice the sample flow of the other two EDI monitors. Nevertheless, the CACE readings among the EDI monitors only deviate very little. In general, little flow dependence of the EDI signal has been observed so far in the laboratory.

Samples for IC measurements were taken before the addition of chloride and at every dosing step. The water was

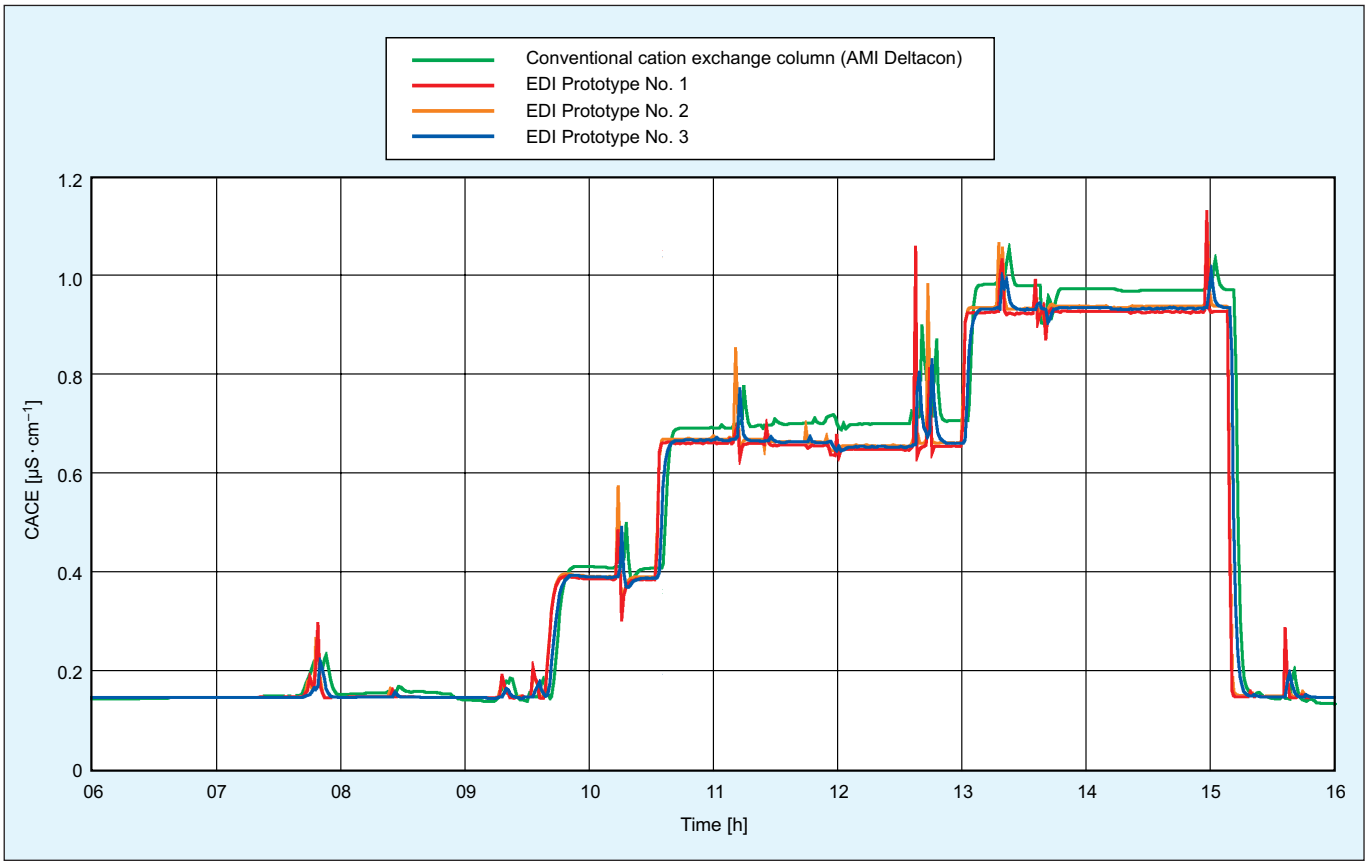

Figure 4:

CACE during chloride dosing analyzed by ion chromatography. CACE measurements after a conventional cation exchange column (AMI Deltacon, shown in green) and with three prototypes using EDI (shown in red, orange and blue) are plotted. No ammonia or other alkalizing agents were dosed. Three different concentrations of sodium chloride were added, corresponding roughly to 20 µg · kg^{-1}, 45 µg · kg^{-1} and 70 µg · kg^{-1} of chloride. An offset of 80 nS · cm^{-1} was added to the EDI measurements to adjust the baseline CACE for better visual comparison. This offset is however not sufficient to correct for the increasing difference between both techniques with increasing chloride concentration. Spikes in the data correspond to instabilities in the flow in the dosing system and are correctly detected by all monitors. The sample flow was adjusted to 6 L · h^{-1} for the Deltacon and the EDI monitor shown in orange (to have similar conditions for the IC comparison below) and to 3.5 L · h^{-1} for the two other EDI monitors.

sampled directly from the inlet and outlet stream via a constant head. Water for cation and anion chromatograms was sampled simultaneously. A Metrohm Compact IC flex was used and a sample volume of 7 mL was injected. As an example the chromatograms sampled with 20 µg · kg^{-1} chloride are shown in Figure 5. The measured concentrations together with the experimental conductivities are listed in Table 3.

No sodium could be detected at the outlet of either the cation exchange column or the EDI module, showing that also the EDI device is able to eliminate all cations. The chloride concentration is always the same at all three locations sampled. So neither cation leakage through the EDI nor chloride loss can explain the deviation of the two measurements. In fact, the only difference in the ion chromatograms between the EDI and the cation exchange column is a more pronounced sulfate and, interestingly, also formate leakage from the cation exchange resin of the passive column.

A calculation of the expected conductivities from the ion concentrations measured revealed that the sulfate and formate leakage of the resin is not strong enough to explain the high baseline CACE, especially for the conventional cation exchange column. A CACE reading of 63 nS · cm^{-1} would be expected based on the IC measurements instead of the experimental value of 140 nS · cm^{-1}. Table 4 lists the results of these calculations for all chloride concentrations, and the differences between the experimental and calculated values are plotted in Figure 6 (in blue).

In the presented experiment the baseline CACE of the passive cation exchange column is relatively high due to a low sample flow through the column and fresh resin (running time about 10 days). These conditions were chosen on purpose in order to make it easier to track the reasons for the offset. However, even under ideal conditions with a well-washed resin, it is hard to achieve a baseline CACE below 100 nS · cm^{-1}. Assuming the same or less sulfate and formate leakage, there is still a difference of roughly 40 nS · cm^{-1} to the calculated value.

In general, it can be observed that the experimental conductivities measured with the passive exchange column are considerably too high compared to the calculated ones including only formate and sulfate ions. So there must be another anionic contribution invisible to the IC coming from the monitor. There are then two options, either a carbon dioxide ingress through the walls and fittings of the cation exchange column or organic anions with too long retention times on the column to be detected by the IC. It is known that polystyrene resins as used in

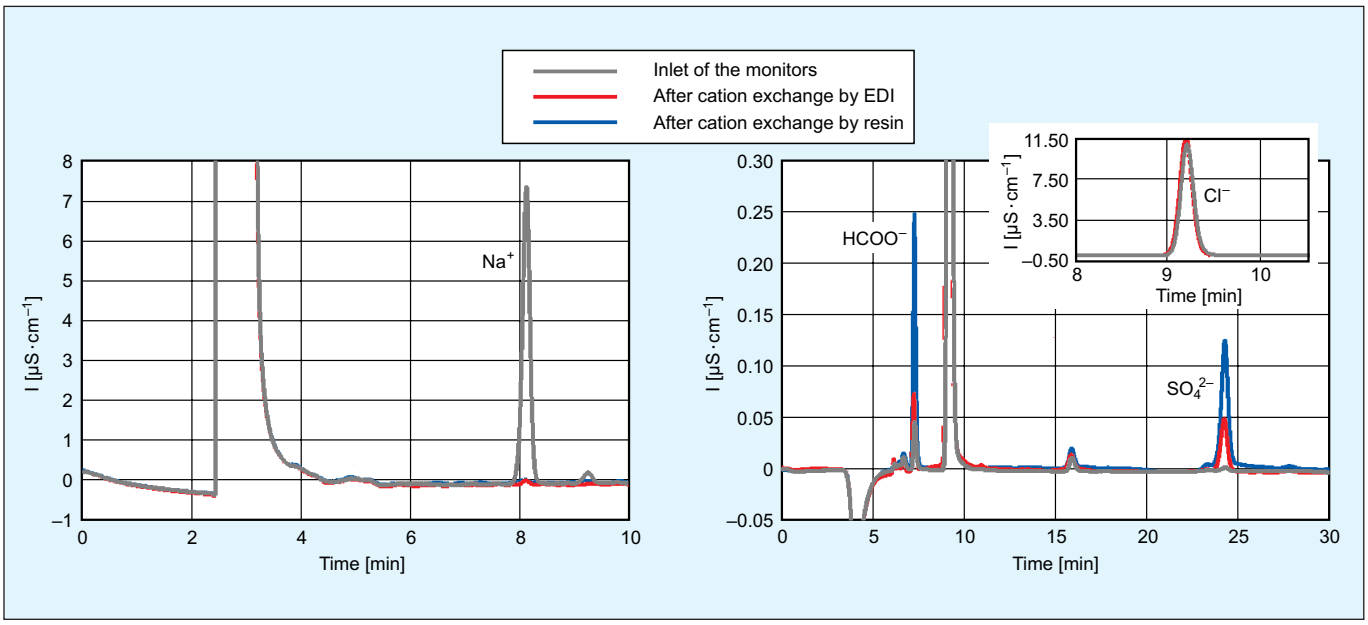

Figure 5:
Ion chromatograms recorded at the inlet of the monitors (grey), where the specific conductivity is measured, and after the cation exchange, where the CACE is measured. Cation exchange was achieved either by EDI (red) or by a cation exchange column (blue). Samples for the cation chromatograms (on the left) and the anion chromatograms (on the right) were taken simultaneously. The chromatograms shown correspond to the first dosing step, so approximately 20 µg · kg^{-1} of chloride. As can be seen from the cation chromatograms, no sodium leakage is observed. The chloride concentration detected is the same before and after the cation exchange. However, sulfate and formate leakage from the resin is more severe for the conventional cation exchange compared to EDI due to the larger volume of resin used.

	Conductivity [nS · cm^{-1}]	Concentration [µg · kg^{-1}]			
		$HCOO^-$	SO_4^{2-}	Cl^-	Na^+
Ultra-pure water					
Inlet	56	0.2	0.0	0.0	0.0
Outlet resin	140	0.9	1.2	0.0	0.0
Outlet EDI	65	0.3	0.2	0.0	0.0
20 µg · kg^{-1} Cl^-					
Inlet	140	0.2	0.0	21.0	14.3
Outlet resin	400	0.8	1.1	21.8	0.0
Outlet EDI	310	0.3	0.4	21.4	0.0
45 µg · kg^{-1} Cl^-					
Inlet	230	0.2	0.0	48.2	31.7
Outlet resin	700	1.2	1.1	47.5	0.0
Outlet EDI	590	0.2	0.2	47.6	0.0
70 µg · kg^{-1} Cl^-					
Inlet	300	0.2	0.0	70.2	47.0
Outlet resin	980	1.0	1.5	69.4	0.0
Outlet EDI	860	0.3	0.3	69.8	0.0

Table 3:
Measured conductivities and ion concentrations.

	Conductivity [nS · cm^{-1}]			
	Experimental	No Additional Anion	With Benzene Sulfonic Acid	With Carbon Dioxide
Ultra-pure water				
Inlet	56	55	55	55
Outlet resin	140	63	141	139
Outlet EDI	65	57	65	61
20 µg · kg^{-1} Cl^-				
Inlet	140	134	134	134
Outlet resin	400	292	396	254
Outlet EDI	310	278	297	284
45 µg · kg^{-1} Cl^-				
Inlet	230	231	231	231
Outlet resin	700	615	720	654
Outlet EDI	590	600	619	604
70 µg · kg^{-1} Cl^-				
Inlet	300	311	311	311
Outlet resin	980	885	991	916
Outlet EDI	860	871	890	873

Table 4:
Experimental conductivities and calculated conductivities assuming different models.

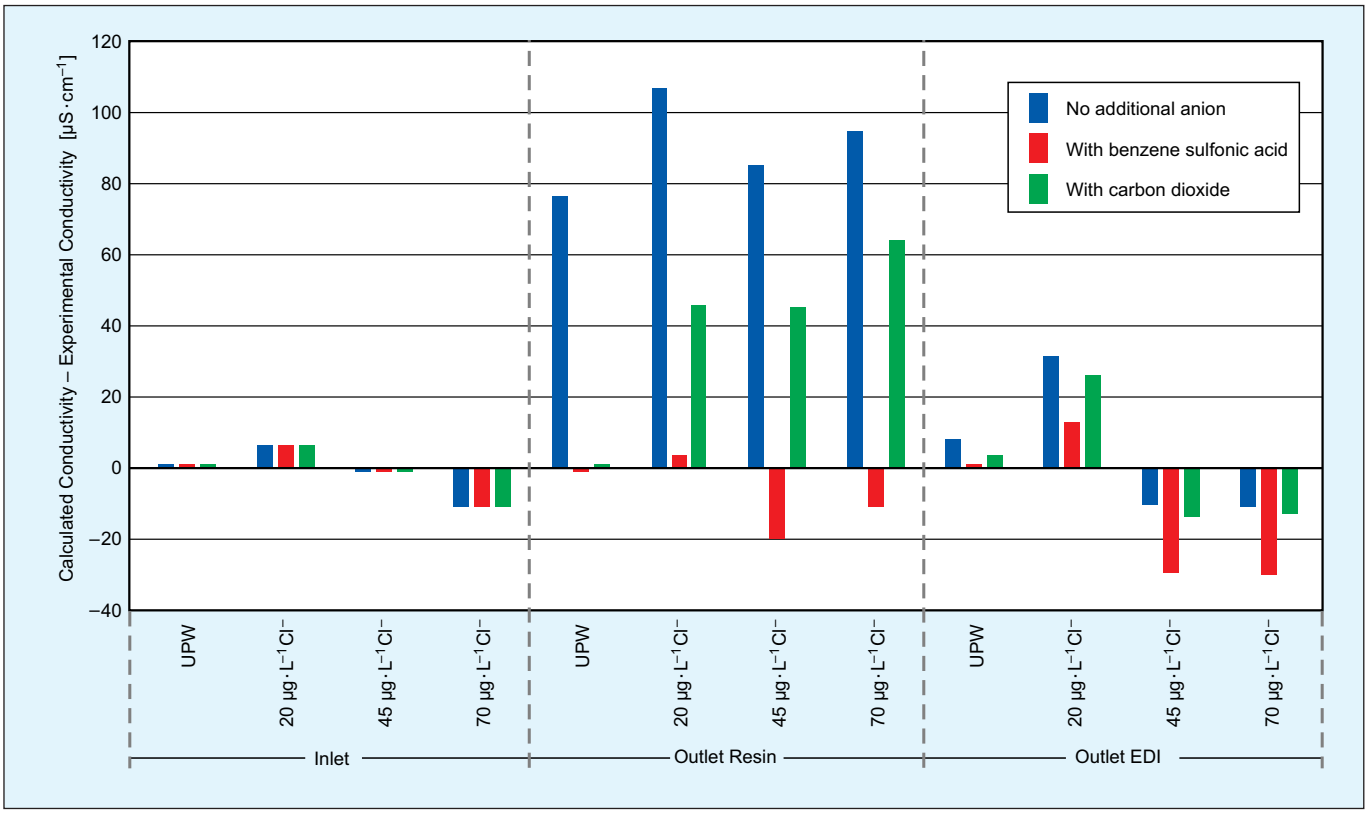

Figure 6:
Difference between the experimental and the calculated conductivities for the three different models. The conductivity at the inlet of the monitors is reproduced very well in the calculations from the IC concentrations and is also not affected by the model chosen. The conductivities after the cation exchange column are however too high when only the formate and sulfate concentrations measured are included in the calculation (shown in blue). So an additional contribution from another strong acid leaking from the resin or carbon dioxide needs to be considered. Of these two options only the model with a benzene sulfonic acid (shown in red) can explain the measured conductivities. The CACE measured with the EDI modules (shown on the right) can be fairly well explained with the IC results. As the offset for the ultra-pure water (UPW) measurement is low, adjusting the UPW value with either benzene sulfonic acid or carbon dioxide does not change the calculations much.

this study can release polystyrene sulfonic acids. Both options were calculated to see if these models are able to explain the measurements. For the calculation the concentration of carbon dioxide or benzene sulfonic acid (as an example of a simple aromatic sulfonic acid) was adjusted such that the CACE without chloride dosing could be explained. This concentration was then kept constant for all four calculations. The results are summarized in Table 4 and Figure 6.

Of these two models only the benzene sulfonic acid is able to explain the high experimental CACE values. The carbon dioxide model fails to reproduce the conductivities as soon as chloride is added. This can be explained by the fact that pH at the outlet of the resin is lower the more chloride is present. Therefore, the equilibrium between carbon dioxide and hydrogen carbonate shifts more and more towards carbon dioxide, not contributing to the conductivity. In the aromatic sulfonic acid model, a constant leakage of 0.28 µmol · L^{-1} (or 44 µg · kg^{-1}) of benzene sulfonic acid would be necessary to explain the observed conductivities. These numerical values change of course if another sulfonic acid or a mixture is assumed in the model, but the overall results are the same. This model of sulfonic acid leakage might also be able to explain the temperature dependence of the CACE readings observed in Figure 3. A higher sample temperature should lead to more sulfonic acid leakage, probably caused by the higher mobility of the ions within the resin. To experimentally identify and quantify the observed anion leakage further experimental work is required.

The same calculations as for the passive ion exchange were done for the EDI module. As the baseline CACE after EDI was already very close to the optimal value of 56 nS · cm^{-1}, almost no additional anions had to be assumed in the model calculations. So there is no difference between the three cases within the accuracy of the measurements and calculations. The contamination through the EDI module is considerably less than through the cation exchange resin, making the results more straightforward to interpret.

The fact that an offset caused by anions leaking from the resin does not lead to a constant offset to the theoretical value might seem counterintuitive at first sight. However, one needs to keep in mind that the total ion concentrations are still very low, so that the contributions from the hydroxide ions cannot be neglected. Close to neutral pH, the dosing of sodium chloride not only leads to a generation of protons, but also to a consumption of hydroxide ions. So the conductivity rises less compared to the same amount of sodium chloride dosed to an acid solution. In theory, in the experiment described above the pH at the outlet of the resin should be pH 7 without chloride and pH 5.7 with 70 $\mu g \cdot kg^{-1}$ chloride. However, the anions leaking from the resin cause a slightly acidic pH at the outlet (pH 6.5 for benzoic acid without chloride) and therefore a steeper increase of the CACE with the chloride concentration as naively expected. Only once the change in hydroxide ion concentration upon addition of chloride can be neglected does the offset converge to a constant value.

CONCLUSION

Already from this simple laboratory experiment it is obvious that there is a contribution to CACE coming from the measurement itself, more precisely from the cation exchange resin used. Though the effects described in this paper might be considered minor in practice and only interesting in the laboratory environment, they should also be noticeable in a power plant, especially when a low baseline CACE can be achieved.

The amount of anionic impurities from the resin not only influences the baseline CACE but also the response to anion ingress, at least as long as the total anion concentration is low. This highlights once again the difficulty in correctly interpreting the CACE values, their being a sum parameter over all ionic species present. Unfortunately, the precise chemical nature of all anions leaking from the resin could not be determined yet. Their amount seems to be proportional to the effective contact time between resin and sample.

Using EDI instead of the passive cation exchange column gives less contribution from the resin to the CACE reading, resulting in lower and more correct CACE values. No cation leakage through the EDI module nor chloride loss could be detected, making the EDI technique suitable for use in CACE monitors. As the impact of the resin on the measurement is minimized, temperature-dependent anion leakage and the flow dependency of the measurements are also minimized, leading to very reproducible measurements.

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Designed, manufactured and tested in Switzerland

We provide process-oriented modular solutions; designed by us, built for you, dedicated to your requirements.