

A Q&A

Perchlorate Analysis by IC: Water Impact



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Perchlorates are found in many commercially-available products and have been identified as an environmental pollutant. Since most perchlorates are water soluble, they enter potable water supplies quite easily. This Q&A session with Joseph Plurad, the North American Field Marketing Manager for Milli-Q Lab Water Systems at MilliporeSigma discusses the problems associated with measuring perchlorate levels and what water purification technologies are available to provide the highest quality water for ion chromatography.

Why is the analysis of perchlorates of interest to our audience today and why did Millipore Sigma choose to investigate this?

Over the last 20 years, perchlorates have received a lot of attention from regulatory agencies because they've been identified as an environmental pollutant with real impact on human health, which can interfere with iodide uptake by the thyroid gland. One of the most recognized sources of perchlorate contamination is from rocket fuels and other propellants. Other sources also include fireworks, matches, disinfectants and some bleaching agents and some fertilizers made from sodium nitrate deposits. And since most perchlorates are readily water soluble, they enter the groundwater water systems quite easily.

Additionally, back in the late 1990s, the EPA developed a detection method for perchlorates in drinking water using ion chromatography. However, ultrapure water is one of the most important reagents in ion chromatography, which is ultimately derived from local drinking water supplies, and as a result, is likely to already have detectable levels of perchlorate.

When considering reagent water for use in ion chromatography, which specific contaminants have the greatest impact on the analysis?

The water needs to be effectively ion-free. It's best to make sure that the reagents used don't have the ion you're trying to determine. So, ionic contaminants in the water are a potential problem and in particular anything else that would elute near the perchlorate peak.

However, we still need to be concerned about organic contaminants, because they can coat the active surface of the resins in the column. This can result in poor peak resolution, low intensity, and shifting baselines, affecting the ability to accurately identify and quantify the perchlorate ion. Some dissolved gasses are also known to cause problems with IC, but this is typically addressed by degassing the water and eluents prior to use.

Other contaminants that could impact the IC system's performance are particulates that could block columns, leading to an increase in pressure, and potentially damaging the system. And finally, bacteria and other microbials growing in the bottle could contribute other biological byproducts in the ultrapure water.

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What water purification technologies are used to provide the best water for ion chromatography and specifically perchlorate analysis by IC?

The best ultrapure water is made from the local municipal tap water. It's also important not to produce significantly more water than you plan to use in your experiments that day. And by owning the purification process from tap water to final use, lab professionals have full control of the water, resulting in better confidence in its quality.

Generally, we recommend purifying the tap water to an intermediate stage via our Elix electrode ionization process. Tap waters pass through pre-filters to remove large particles, some disinfectants and organics, and to sequester ions that cause hardness, which can generate other purification problems downstream. After pre-filtration, the water passes through a reverse osmosis (RO) membrane that will remove more than 95% of all the contaminants in the tap water. Our patented electrodeionization process will further remove any ionically-charged species from the water resulting in 1-50 megohm resistivity.

After deactivating bacteria with UV radiation, we'll store the water before final cleanup for use. And when the analyst is ready for the ultrapure water for IC, we'll pass it through purification steps embedded in our Milli-Q ultrapure water systems. The water will pass through a dual wavelength UV lamp not only to mitigate microbial risk, but to also photo-oxidize trace organics into free radicals that can be removed with ion exchange media. The ion exchange resin will further remove ions that may have passed through the Elix technology. And the final filtration step is to pass the water through a 0.2 micron filter to remove any remaining particles and microbes that might remain. Using these techniques, we produce 18.2 megohm water with a Total Organic Carbon (TOC) content of < 5 ppb.

What did Millipore Sigma do to test the efficacy of water purification to provide suitable water for perchlorate analysis?

Perchlorates exist in many municipal drinking water sources, so we evaluated the ability of four different Millipore Sigma water systems to remove perchlorate from water. First we tested a Direct-Q 3 UV system utilizing reverse osmosis, UV photo-oxidation, activated carbon and ion exchange. The second system was an Elix 5 system, which combines activated carbon, reverse osmosis and electrodeionization to produce water similar to distilled water. And finally, our two entry-level ultrapure systems, the Simplicity UV and Milli-Q Advantage A10 water systems that use UV photo-oxidation, activated carbon and ion exchange to produce ultrapure water from the Elix 5 water system.

Because the tap water did not have significant perchlorate content, we spiked the tap water fed to the Direct-Q and Elix systems with about 80 ppm of perchlorate. Then the Elix water used to feed the Simplicity and Milli-Q systems was also spiked with perchlorate at an order of magnitude lower than

the tap water (80 and 120 ppb). It's important to emphasize that the level of perchlorate we spiked into each of these systems was significantly higher than you would normally expect to find in tap water, which is typically around 25 ppb. The final step was to measure the perchlorate content by IC at the inlet to each system and after each of the purification steps in the separate lab water systems.

So, can you share some of those results with us?

The reverse osmosis step removed 97% of the perchlorate that was in the tap water fed to it. When the RO water was then purified via traditional ion exchange or via electrodeionization in our Elix module, perchlorate levels already fell below the 5 ppt detection limit. When the RO and the Elix water was then passed through ion exchange resins in the Simplicity system and the Milli-Q system, perchlorate was not detected at the outlet of either system.

So basically, with the exception of reverse osmosis alone, any Millipore Sigma system with ion removal after the RO would be suitable to use. So even though Elix water can produce perchlorate-free water, we still have to be concerned about organic microbial and particular contaminants that could impact the analysis. So for that reason, we would recommend using an ultrapure system like a Milli-Q or Direct-Q system for IC analysis.

Is MilliporeSigma doing anything else to meet the needs of the laboratories requiring ultratrace analysis of ions?

In the last 20 years since the EPA methods first came out for perchlorate analysis, there have been continual advancements in detection technologies which are achieving lower detection limits allowing for smaller sample sizes, and even the elimination of some sample preparation steps. Additionally, from an analytical perspective, 18.2 megohm ultrapure water can still have ionic contaminants that cannot be quantified using resistivity measurement. For that reason, we have a responsibility to our customers to provide water that meets these strict requirements. Our ultrapure systems are highly customizable at the dispensing point to meet the demands of today's analytical scientists, whether they are ionic, organic or microbial in nature.

One example of this would be our Q-POD Element dispensing unit. It was originally intended for ICP-MS applications for use in a clean room and as a result uses semiconductor-grade materials in its construction. But inside this Q-POD Element is an additional ion exchange cartridge, as well as low-leeching materials that minimize recontamination along with a 0.01 micron semiconductor-grade filter for further particle removal. The original target of this Milli-Q accessory was for trace metals but we can take advantage of the design and the purification media to achieve low ppt ionic levels, even though resistivity meter will continue to report 18.2 megohm.