

# Ultrapure Water Dechlorination Addressing GAC Bed Chlorine Leaks

# Introduction

Ultrapure water (UPW) used for semiconductors, chemicals, or beverage manufacturing has strict limits on the presence of either oxidants or reducers, and dechlorination is frequently done with granulated activated carbon (GAC) adsorbing chlorine, either followed by reverse osmosis (RO) membrane filtration or not. Whether dechlorinating via adsorption or chemical addition (sulfite-based reagents), regardless of the use of RO membranes downstream, disinfectant residual in production water must be thoroughly monitored and controlled. Currently, such monitoring and proportional addition-reduction potential (ORP) measurement. Measuring chlorine residual with direct colorimetric analysis has many advantages over e-chem methods, allowing to provide accurate results and differentiation between chlorine species should it be necessary. For example, total chlorine analysis allows the measurement of free chlorine only, or of chloramines, or of all chlorine species, or along with other oxidants present in the water (TRO = total residual oxidant). All these oxidants may be detrimental to the RO membranes, the produced water, or the manufactured goods. Therefore, total chlorine measurements provide the necessary assurance in any membrane protection applications, especially after adsorption-based dechlorination, which can create various chloramines, the presence of which may be missed by free chlorine only methods.

### Problem

GAC is used in the UPW preparation cycle for electronics or pharmaceutical manufacturing, as well as in beverage production, to name just a few industries. The effect of channeling, when carbon beds (GAS tanks) develop paths between granules saturated with contaminants that are adsorbed and therefore pass the chlorine through, is well-known in the industry. Since replacement or regeneration of the activated carbon is normally done on a schedule, detecting chlorine leak is difficult without active measurement. Intermittent grab sample analysis may help, however, not all facilities use appropriate methods and equipment, and the leaks often happen undetected until it is too late. Therefore, timely detection of chlorine leak through the GAC beds is an issue that needs to be resolved.

## Solution

Ultra-Low Range (ULR) CL17sc chlorine analyzer uses colorimetric method to measure total chlorine residual with unparalleled accuracy provided by low limit of detection (LOD) of 8 ppb. This means that the readings above ~3 x LOD = LOQ (limit of quantitation) are accurate within +/- 8 ppb or 5%, whichever is greater. Therefore, accurate chlorine readings provided by the analyzer every 150 seconds (2.5 minutes) will show a complete picture of the dechlorination process, detect any excursions of chlorine above the set level, and help to manage GAC tanks/beds properly. All this will help to ensure specified quality of RO feedwater, of produced UPW, and thus the quality of the manufactured goods.



Ultra-Low Range (ULR) CL17sc chlorine analyzer

#### **Case Study**

A test was conducted at a microelectronics manufacturing facility with primary objectives to improve the efficiency of their dechlorination process while reducing the risk of chlorine damage to the first stage RO membranes. The plant has several RO racks containing over 200 individual cartridges with GAC pretreatment and addition of metabisulfite (MBS) to destroy extra chlorine residual in the RO feedwater as necessary, should chlorine break through the GAC beds. Around 30 membrane cartridges are replaced during a typical year, which is approximately \$10,000, including the costs of membranes, labor, and lost revenue. Every three years on average, the RO membrane users must run an autopsy of failed membranes, which is usually done by contractors, and it can cost a few thousand dollars extra. Therefore, any premature failure of RO membranes due to chlorine breakthrough is a costly problem.

The facility monitored chlorine using grab sample analysis before ULR CL17sc analyzer was installed at the first pass RO system influent line, after the GAC beds and MBS injection point, with the source water (city tap water) containing 3–4 ppm total chlorine. The analyzer had been tested for several weeks to validate its accuracy and response time before a long-term test during which it detected an event related to a GAC tank failure, Figure 1.

The RO feed comprised the combined effluent from four carbon beds/tanks. Two out of four tanks account for ~20 % of the total flow each and the other two for ~30 % each. The event presented in Figure 1 happened after

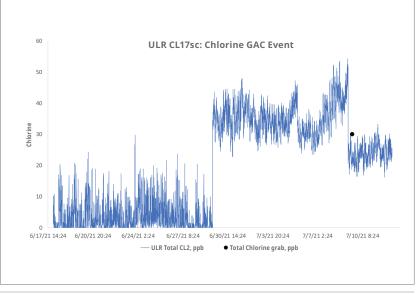


Figure 1 – GAC failure detected by the tested ULR CL17sc and verified with a reference analysis after the root cause was found and eliminated.

the MBS feed was deemed unnecessary and stopped. It was discovered that one GAC tank's effluent was contributing 150 ppb of chlorine to the combined sample. This contribution was immediately detected and recorded by the analyzer, while was missed by the personnel during the holiday week (grab sample analysis only). Once the exhausted media was replaced, the chlorine concentration came down to the desired level of < 30 ppb as was confirmed by the grab sample analysis, Figure 1. The test demonstrated that GAC media can be exhausted or develop channels passing chlorine through.

#### Outcomes

During the ULR CL17sc testing at this and many other municipal and industrial facilities, the analyzer demonstrated expected sensitivity to sulfite feed rate changes and good accuracy against grab sample analyses (e.g. Method 8167, DR3900).

The results of this and many other case studies revealed the value of highly accurate direct chlorine measurements at minimal maintenance efforts supporting all chemical and labor cost savings elucidated by the instrument and projecting the full ROI in two years on average.



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