Pilot Testing of New WBA and SBA Resins for Chromium-6 Removal

Report

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TABLE OF CONTENTS

Executive Summary

A new maximum contaminant level (MCL) of 10 ppb for chromium-6 became effective in California in July 2014. Weak-base anion exchange (WBA) and strong-base anion exchange (SBA) are two of the best available technologies. The City of Glendale has tested three WBA resins in the past, which all had a high Cr(VI) capacity but each has challenges. SBA resins were also tested at Glendale in previous studies. All the resins had relatively short operational life before regeneration is required. AquaNano LLC has two new resins under the trademark of Captymer® (one WBA, AQ208-WB and one SBA, AQ60-MP) that are in the development stage and showed promising performance in their bench-scale studies. A third-part and independent pilot study was conducted by Hazen and Sawyer at Glendale to evaluate the two new resins performance for Cr(VI) removal using the same methodologies as used for testing other resins. In addition, the resins were also evaluated for potential chemical leaching and spent resin characteristics for disposal.

The two new resins were tested using a pilot skid that has been successfully used for previous resin testing. WBA was tested with 2-minute EBCT for six months and then reduced to 1-minute EBCT to maximize bed volumes during the test period. SBA was tested with 2-minute and 4-minute EBCT as a single pass without regenerations. WBA received a stream of Glendale's GS-3 water with carbon dioxide (adjusted pH to a target of 6.0) that was treated by the existing demonstration-scale WBA process. SBA received GS-3 raw water without pH adjustment. The field testing last for nine months. Details of the testing methodologies are discussed in Section III Testing Approach.

Results of this study showed the new WBA resin had a high Cr(VI) capacity. Cr(VI) in the new WBA resin effluent was < 0.02 ppb until approximately 92,000 BVs. At that time, the EBCT was decreased from 2 minutes to 1 minute to maximize the bed volumes during the test period. The effluent Cr(VI) concentration slightly increased after the EBCT change and fluctuated in the range of <0.02 – 1.5 ppb. At the end of the field testing, Cr(VI) in the WBA resin effluent was 0.36 ppb at approximately 198,600 BVs. Cr(VI) in the 50% port samples was 6.4 ppb at approximately 397,000 BVs. The results suggest initial breakthrough occurred at approximately 98,200 BVs, which was possibly accelerated by the shorter EBCT, considering the slow rate of Cr(VI) removal by WBA in general. Compared with the other two WBA resins (A and B) tested at Glendale during 2012-2013, the new WBA resin did not show an initial Cr(VI) leakage observed with Resins A and B. The influent Cr(VI) concentration was slightly lower than tested for Resins A and B, although it was the same water source. The impact of influent Cr(VI) level on resin life has not been quantified. However, a Water Research Foundation study suggests the impact of Cr(VI) concentration on WBA resin life was minor (Najm et la., 2014). This study results suggest the new WBA resin likely has a greater Cr(VI) capacity compared with Resins A and B as well as PWA7.

The new WBA resin removed alkalinity, nitrate, sulfate and phosphate, although fast breakthrough occurred (less than one month – by the second sample collected). Uranium was

effectively removed throughout the 9-month test period. Resin effluent pH was noted reduced significantly on the first day of testing, which returned to the influent pH in less than three days. A total of 172 organic compounds were tested at startup, midpoint after a shutdown and restart, and at the end of testing (without a shutdown) to ensure that treatment dose not introduce another contaminant of concern like the PWA7 resin leaching formaldehyde. Nitrosamines, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), aldehydes and ketones leaching was either not detected or at levels similar to the raw water (except 1,3-dichloropropane). Formaldehyde was not detected as observed with PWA7. The spent WBA resin tested in this study was characterized as a non-RCRA hazardous waste (hazardous in California) due to chromium concentration above the Total Threshold Limit Concentration (TTLC) limit. In addition, uranium was above the regulatory limit for low level radioactive material. At full-scale, spent resin would need to be disposed to a non-RCRA hazardous waste landfill that also accepts the uranium concentration. Absorbent materials may be used in disposal, which would reduce uranium concentration. However, landfills should be consulted for their specific requirements and limits on acceptable uranium concentrations.

The SBA results suggest significantly greater Cr(VI) capacity compared with other SBA resins tested at Glendale during 2012-2013 as well as other previous studies. The test water contained relatively high sulfate (110 – 120 mg/L), which is expected to negatively impact SBA resin life. Resin effluent Cr(VI) reached 8 ppb at approximately 12,000 to 14,000 BVs (for the two columns tested), which is about 17 to 19 operational days with a 2-minute EBCT. By comparison, the other SBA resins had operational life of 4 to 5 days when tested with the same water. A greater EBCT (4 minutes) did not demonstrate improved Cr(VI) capacity or resin life for the new SBA resin.

Simultaneous removal of other constituents, including alkalinity nitrate, sulfate, and phosphate were observed but with faster breakthrough than Cr(VI) (less than three days – by the second sample collected). Uranium was effectively removed throughout the 9-month test period. Resin effluent pH was reduced to 6.3 (compared to the influent pH of 7.2) on the first day of testing, and then returned to the influent pH level in less than three days. Constituents leaching testing revealed the majority were either not detected or at lower levels than in the raw water. Four organic compounds were detected at startup or midpoint after a shutdown and restart, including 2-butanone (46 µg/L), propanal (14 µg/L), glyoxal (11 µg/L), and methyl glyoxal (14 µg/L). None of them have a regulatory limit. The spent resins in this study were characterized as a non-hazardous waste by federal and California standards. Uranium concentration of the spent resin with 2-minute EBCT was below the regulatory limit for the conditions tested, although it is still considered TENORM. Disposal options depend on landfill-specific limits. The uranium concentration in the spent resin is affected by raw water uranium and resin operational life. Thus, it may increase or decrease for some water agencies. Resin regeneration was not tested in this study. It is recommended to evaluate the resin performance with regeneration, especially when this new resin is considered for full-scale application with regenerations.

I. Introduction

A new maximum contaminant level (MCL) of 10 ppb for chromium-6 in drinking water became effective in California in July 2014. In anticipation of this regulation, the City of Glendale, California has been conducting nearly a decade-long research program on Cr(VI) removal options. Three primary technologies have been shown to achieve levels below the MCL, including: weak-base anion exchange (WBA), strong-base anion exchange (SBA), and reduction coagulation filtration (RCF).

Glendale has tested three WBA resins in previous research program. Each has a high capacity for Cr(VI), but each has challenges. For example, the Dow PWA7 resin initially leaches formaldehyde above the California Notification Level of 100 ppb unless conditioned, and the resin also accumulates uranium so that the resin runs the risk of being classified a low-level radioactive waste. Two newer resins, ResinTech SIR-700 and Purolite S106, do not leach formaldehyde, but experience an initial period of incomplete chromium-6 removal in some water qualities.

Four SBA resins have also been tested at Glendale for Cr(VI) removal, providing information on the resin life before regeneration is required.

AquaNano developed two new resins (one WBA and one SBA), which have undergone bench-scale testing using water with key constituents similar to Glendale. The resins differ in the use of a new polymer matrix, reportedly offering a structure with higher total exchange site capacity.

The AquaNano resins are reported[1] to offer potential advantages compared with currently available resins, including:

- SBA: AQ60-MP – Three times the capacity of Dow SAR and Dow 11
- WBA: AQ208-WB – Less leakage observed for initial period compared with SIR-700 and no formaldehyde leaching

[1] Communications with Han-Ting Chang.

II. Objectives

The objectives of the project were as follows:

- Provide a third-party, independent evaluation of the two new resins for Cr(VI) removal.
- At pilot-scale, determine the effectiveness of the resins for Cr(VI) removal in Glendale water using the same methodologies as used for testing other resins.
- Evaluate potential chemical leaching from the resins and characterize spent resins for disposal.
- Evaluate whether the new resins offer significant advantages to the other resins tested to date.

III. Testing Approach

This section describes the testing approach, including pilot unit and operational conditions, test water quality, sampling locations and schedule, analytical methods, quality assurance and quality control.

Pilot Unit and Operational Conditions

Glendale has effectively used ion exchange column skids at the GS-3 well site for several years to test WBA and SBA resins, and shown the configuration to accurately reflect full-scale performance. The skid includes coarse filtration to remove sand and minimize the need for backwashing, 2.5 inch diameter downflow resin columns with a screened sampling port at 50% bed depth, a flow meter and totalizer on each column. Two cartridge filters, 1 µm followed by 0.5 µm pore size, were installed for this pilot to remove fine particles which were observed in the demonstration-scale testing at the same well site. A photograph of the ion exchange skid used in this project is shown in Figure 1.

Figure 1. Photo of the Pilot Unit

Table 1 shows the operational conditions used for testing the WBA and SBA resins. A resin bed depth of 13 inches was used for the WBA resin, which provides an aspect ratio (height to column width) of 5. A resin bed depth of 30 inches was used for the SBA resin, which provides an aspect ratio of 12. One manufacturer recommends a minimum bed diameter of approximately 0.75 inches and an aspect ratio of at least 4 (Dow, 2011). A column diameter to resin bead diameter ratio of approximately 127 (bead diameter of about 500 micron) was used. Most literature reports that a ratio of 50 to 100 is effective in avoiding wall effects in filtration pilot columns (AWWA, 1982; Kawamura, 2000). However, two other tests indicated that wall effects may not be observed down to 26:1 (McLellan et al. 2011; Lang et al., 1993). For testing in Glendale, a column diameter to resin bead diameter of approximately 40 was proven to be effective at representing full-scale bed life.

The WBA resin was tested with 2-minute empty bed contact time (EBCT), which was tested for PWA7, SIR-700 and Purolite S106 during the pilot in 2012 and 2013 (Blute et al. 2014). The WBA EBCT was decreased to 1 minute at approximately 92,000 BVs (six months after startup) to maximize the number of bed volumes during the limited test period.

The SBA resin was tested with 2-minute EBCT, which was the EBCT tested in Glendale at a pilot scale (McGuire, et al., 2006) and an ongoing pilot at Coachella Valley Water District for other SBA resins. The same SBA resin was also tested with 4-minute EBCT as laboratory testing by AquaNano scientists indicated that a greater EBCT may improve SBA resin life.

Table 1. WBA and SBA Resins Operational Conditions

Design Parameter	**WBA:** **AQ208-WB**	**SBA:** **AQ60-MP (Lower EBCT)**	**SBA:** **AQ60-MP (Higher EBCT)**
Column Diameter (in)	2.5	2.5	2.5
Cross Sectional Area (sf)	0.034	0.034	0.034
Bed Depth (in)	13	30	30
Bed Volume (cf)	0.04	0.09	0.09
Empty Bed Contact Time (min)	2.0*	2.0	4.0
Flow Rate (gpm)	0.14	0.32	0.16
Hydraulic Loading Rate (gpm/sf)	4.0	9.5	4.75
Service Flow Rate (gpm/cf)	3.7	3.7	1.85
Daily Water Required (gal)	196	466	233
BVs per Day per Column	720	720	360
Operating pH Target	6.0	Raw water pH	Raw water pH
Operational Mode	Down flow	Down flow	Down flow
Run Time	9 months	9 months	7 months
Backwash Frequency	Not performed. Not needed.		

*WBA EBCT was decreased from 2 minutes to 1 minute at approximately 92,000 BVs to accelerate throughput in the available time for the study.

Test Water

The pilot study was conducted using Glendale GS-3 well water. At this site, a demonstration-scale WBA system (425 gpm) has been operational since 2010. Carbon dioxide was injected to raw water to provide the low pH required for WBA. For this pilot study, a stream of the pH adjusted water from the demonstration process (post bag filters) was used to test the new WBA resin and the GS-3 raw water was used to test the new SBA resin.

Table 2 summarizes the raw water quality (prior to the cartridge filters) for the SBA resin during this pilot study. The Cr(VI) concentration was in the range of 16 to 21 ppb with an average of 19 ppb. Total Cr concentration was between 17 and 22 ppb, with an average of 19 ppb. Alkalinity was relatively high compared to most groundwaters in California, requiring a high carbon dioxide dose for pH reduction to 6. Chloride and nitrate concentrations were moderate (75 mg/L and 7.7 mg/L as N, respectively). The sulfate concentration was high (117 mg/L), which was expected to negatively affect the SBA resin life. The uranium concentration was between 2.0 and

3.9 pCi/L with an average of 2.9 pCi/L. The raw water pH (without carbon dioxide) was between 6.5 and 7.6 according to field testing, although the high end likely reflects carbon dioxide offgassing of groundwater resulting in an increase in pH.

Table 2. SBA Test Water Quality during the Pilot Study (Prior to the Cartridge Filters)

Parameter (unit)	**Average**	**Range**
Cr(VI) (ppb)	19	16 – 21
Total Cr (ppb)	19	17 – 22
Alkalinity (mg/L as $CaCO_3$)	206	190 – 230
Calcium (mg/L as Ca)	98	89 – 100
Chloride (mg/L)	75	70 – 77
Conductivity (µs/cm)	933	920 – 950
Nitrate (mg/L as N)	7.7	7.3 - 7.9
Phosphate (mg/L as PO_4)	0.15	0.13 - 0.17
Silicate (mg/L)	39	36 – 43
Sulfate (mg/L)	117	110 – 120
Total Organic Carbon (mg/L)	0.42	0.34 - 0.52
Total Suspended Solids (mg/L)	< 10	< 10
Uranium (pCi/L)	2.9	2.0 - 3.8
Uranium (µg/L)	4.3	3.0 – 5.6
pH – Field	7.1	6.5 - 7.6
Temperature – Field (^{0}C)	21.9	19.4 – 25.5

The effects of the cartridge filters on Cr(VI) and total Cr was evaluated by paired sampling from pre- and post-cartridge filters. The results are summarized in Table 3. The samples at pre- and post-cartridge filters contained similar Cr(VI) as well as total Cr concentrations, except the samples collected on May 14, 2014. It is suspected the results for the May sample may be caused by analytical issues when inconsistent Cr(VI) and total Cr results between WBA influent samples collected from the pilot and the same water collected from the demonstration-scale were noted during the same period. Overall, the cartridge filters did not remove Cr(VI) or total Cr to a significant level.

Table 3. Paired Cr(VI) and Total Cr Samples Pre- and Post-Cartridge Filters

Sampling Date	Cr(VI)		Total Cr	
	Pre-Cartridge Filter	Post-Cartridge Filter	Pre-Cartridge Filter	Post-Cartridge Filter
5/14/2014	18	13	18	12
7/23/2014	17	19	19	18
7/31/2014	19	20	19	20
8/6/2014	20	18	19	19

Table 4 summarizes Cr(VI), total Cr, field pH and temperature for WBA test water during the pilot study. Cr(VI) and total Cr concentrations were similar to the SBA test water. pH was in the range of 5.5 to 6.3, with an average of 6.0 (which was the target).

Table 4. WBA Test Water Quality during the Pilot Study (after CO_2 and Bag Filters)

Parameter (unit)	Average	Range
Cr(VI) (ppb)	19*	10 – 23*
Total Cr (ppb)	19^	15 – 21^
pH – Field	6.0	5.5 – 6.3
Temperature – Field (^{0}C)	21.5	18.5 – 24.6

*Five samples with Cr(VI) results between 3.7 and 5.6 ppb during April were excluded, which are suspected caused by analytical issues. The same water collected from the demonstration-scale testing contained Cr(VI) between 16 and 20 ppb.

^ Five samples with total Cr results between 2.4 and 4.9 ppb during April were excluded, which are suspected to be caused by analytical issues. The same water collected from the demonstration-scale testing contained total Cr between 15 and 20 ppb.

Sampling Locations and Schedule

Figure 2 shows the sampling locations on the ion exchange skid represented by "IX". Sampling locations for the WBA resin included raw water (before pH adjustment, designated as IX-1), WBA influent (after pH adjustment, IX-2), test column 50% bed depth (IX-3), and test column effluent (IX-4). SBA column testing locations included test column effluent (IX-5) in addition to the common sampling point for raw water (IX-1).

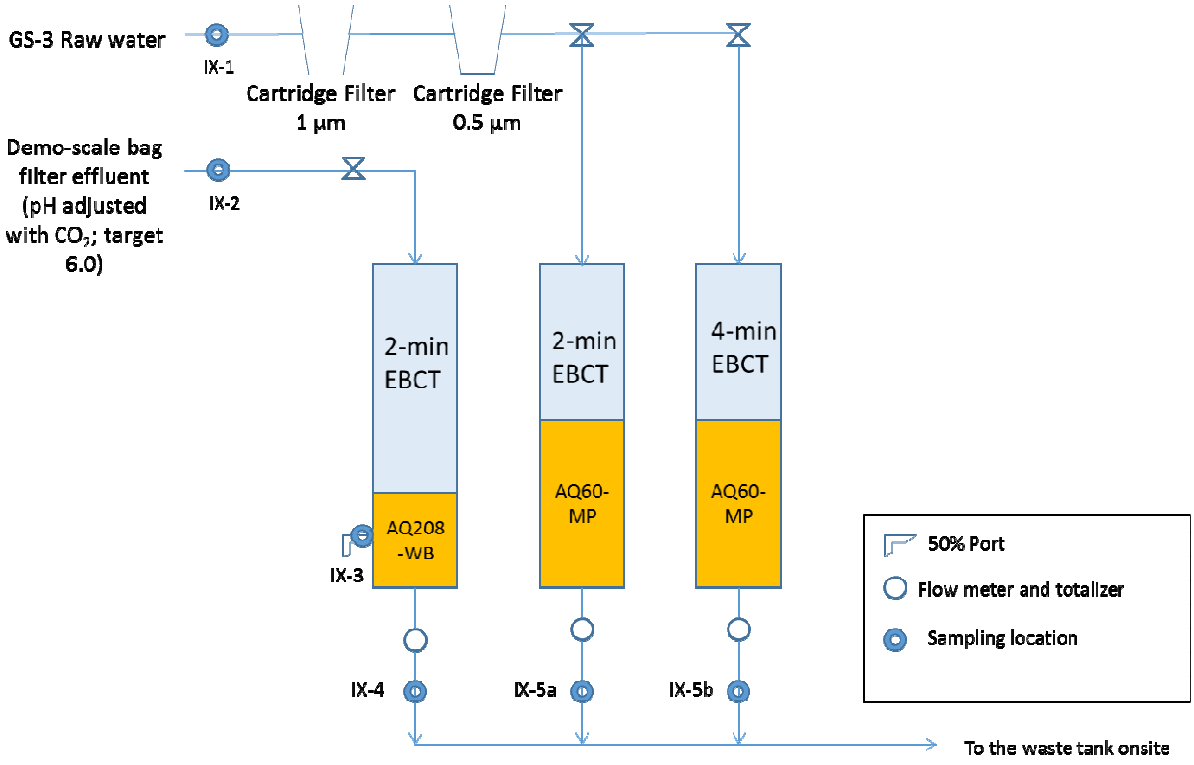

Figure 2. Sampling Locations

The monitoring locations and sampling frequencies for the water quality parameters are summarized in Table 5. Critical water quality parameters that were measured for the ion exchange resins included Cr(VI), total chromium, nitrate, chloride, sulfate, silicate, phosphate, uranium, and pH. Other chemical and physical parameters, including temperature, conductivity, turbidity, total suspended solids (TSS) and alkalinity were routinely measured to characterize water quality. Nitrosamines and formaldehyde, which have been found to leach from ion exchange resins, were measured during startup and midpoint during operation after a start/stop procedure to assess any leaching. In addition, a broad scan for tentatively identified compounds (TICs) for both volatile organic compounds (VOCs) and synthetic volatile organic compounds (SVOCs) was conducted initially and midpoint through operation to ensure that treatment does not introduce another contaminant of concern like the PWA7 resin leaching formaldehyde. Bacti (Total Coliform, *E. Coli* and HPC) was monitored initially and midpoint through operation.

Besides chemical and physical water quality analyses, process-related parameters were recorded to evaluate operations. The process-related parameters included flow rate and numbers of bed volumes of water treated at the 50% port and effluent.

Table 5. Sampling and Analysis Frequency

Analytical Measurement		Monitoring Locations						
		IX-1 (Raw water)	**IX-2 (pH-Adjusted)**	**IX-3 (WBA 50% Port)**	**IX-4 (WBA Effluent)**	**IX-5a (SBA Effluent – 2 min EBCT)**	**IX-5b (SBA Effluent – 4 min EBCT)**	**Residuals Spent Resin**
Routine Laboratory	Cr(VI)	W	W	W	W	3xW	3xW	–
	Chromium, Total	W	W	W	W	3xW	3xW	–
	Alkalinity	W≠	–	–	M	W	–	–
	Calcium	W≠	–	–	M	W	–	
	Chloride	W≠	–	–	–	W	–	–
	Conductivity	M	–	–	M	S, MP	–	–
	Nitrate	W≠	–	–	M	W	–	–
	Phosphate	W≠	–	–	M	W	–	–
	Silicate	W≠	–	–	M	W	–	–
	Sulfate	W≠	–	–	M	W	–	–
	TOC	M	–	–	–	–	–	–
	TSS	M	–	–	–	–	–	–
	Uranium	W≠	–	–	M	W	W	O
Field	pH	W	W	–	W	W	–	–
	Temperature	W	W	–	W	W	–	–
Special Lab	Nitrosamines (8)	–	S*,MP	–	S*,MP	S*,MP	–	–
	SVOCs and TICs	–	S,MP	–	S,MP	S,MP	–	–
	VOCs and TICs	–	S,MP	–	S,MP	S,MP	–	–
	Aldehydes/Ketone	–	S,MP	–	S,MP	S,MP	–	–
	Bacti	S,MP	–	–	S,MP	S,MP	–	–
	TCLP, CWET, Uranium	–	–	–	–	–	–	O

W: Weekly; 3xW: three times a week; M: Monthly.
S: Start-up (once within first 2 days).
MP: Midpoint through test period after a start/stop cycle.
O: Once when spent; resin from each column was mixed and sampled for uranium, TCLP and CWET.
*Nitrosamines were sampled at first flush, after 4 hours, and midpoint through the test period.
≠ Weekly during SBA runs; monthly thereafter to match WBA frequency.
SVOC: semi-volatile organic compounds and tentatively identified compounds (TICs).
TOC: total organic carbon.
TSS: total suspended solids.
VOC: volatile organic compounds.
Bacti: Total Coliform, *E. Coli*, and HPC.
CWET: California Waste Extraction Test (metals only)
TCLP: Toxicity Characteristic Leaching Procedure (metals only)

Analytical Methods

Analytical methods for each water quality parameter are provided in Table 6.

Table 6. Analytical Methods

Analysis	Analytical Method	Analysis Location	Method Report Limit (MRL)
Cr(VI)	EPA 218.6	EEA labs	0.02 ppb
Chromium, Total	EPA 200.8 with digestion	EEA labs	0.2 ppb
Alkalinity	SM 2320	EEA labs	2 mg/L as $CaCO_3$
Calcium	EPA 200.7	EEA labs	1 mg/L
Chloride	EPA 300.0A	EEA labs	1 mg/L
Conductivity	SM 2510B	EEA labs	2 µm/cm
Nitrate	EPA 300.0	EEA labs	0.1 mg/L as N
Phosphate	SM 4500P-E	EEA labs	0.01 mg/L as P
Silica	EPA 200.7	EEA labs	0.5 mg/L
Sulfate	EPA 300.0A	EEA labs	0.5 mg/L
TOC	SM5310C	EEA labs	0.3 mg/L
Uranium	EPA 200.8	EEA labs	1 µg/L or 0.7 pCi/L
pH	SM 4500H+ B	Field	N/A
Temperature	SM 2550	Field	N/A
Turbidity	SM 2130 B	Field	0.02 NTU
Nitrosamines (8)	EPA 521	EEA labs	2 ng/L
SVOCs and TICs	EPA 625	EEA labs	Varies by compound
VOCs and TICs	EPA 524.2	EEA labs	Varies by compound
Aldehydes/Ketones	EPA 556	EEA labs	Varies by compound
Residuals – TCLP	EPA 1311	Lancaster lab	Varies by element
Residuals – CWET	CWET (Title 22)	Lancaster lab	Varies by element
Residuals – Uranium	ASTM5174-91	Lancaster lab	0.05 mg/kg
Total Coliform and E. Coli	SM 9223	EEA labs	1.1 MPN/100ml
HPC	SM 9215B	EEA labs	1 CFU/ml

ASTM: American Society of Testing and Materials
ELAP: Environmental Laboratory Accreditation Program
USEPA: United States Environmental Protection Agency
SM: Standard Methods
EEA: Eurofins Eaton Analytical

Quality Assurance and Quality Control

All laboratory analysis was performed using analytical methods that conform to EPA guidelines and recommended test methods, including those in Standard Methods for the Examination of Water and Wastewater (APHA, 1999). Standard Operating Procedures (SOPs) was be used for all measurements.

Evaluation of the effectiveness of the ion exchange resins depends largely on Cr(VI) and total Cr analyses of the ion exchange effluent. Previous WBA testing found that Cr(VI) is reduced to Cr(III) by the high capacity WBA resins. It is important to ensure that total Cr is removed from the water since Cr(III) can re-oxidize to Cr(VI) in the distribution system. As the critical parameters in evaluating the success of the project, Cr(VI) and total Cr concentration data were subjected to paired sample analyses (i.e., Cr(VI) and total Cr samples collected at the same time). Paired samples were used to assess the chromium speciation.

IV. Testing Results

The test results for WBA and SBA are discussed separately in this sections below.

WBA Results

The WBA resin was evaluated for removal of Cr(VI) and total Cr, removal of other constituents, leaching of other constituents at startup and after a shutdown, and spent resin characteristics for disposal.

Chromium Removal

The Cr(VI) breakthrough curve for the new WBA resin is shown in Figure 3, compared with two other WBA resins tested in previous studies. Cr(VI) in the new WBA resin effluent was <0.02 ppb until approximately 92,000 BVs. At that time, the EBCT was decreased from 2 minutes to 1 minute to maximize the number of bed volumes that could be achieved during the test period. The effluent Cr(VI) concentration slightly increased after the EBCT change and fluctuated in the range of <0.02 – 1.5 ppb. At the end of the field testing, Cr(VI) in the WBA resin effluent was 0.36 ppb at approximately 198,600 BVs. The results suggest initial breakthrough occurred at approximately 98,200 BVs, which was possibly accelerated by the shorter EBCT, considering the slow rate of Cr(VI) removal by WBA resins in general. The Cr(VI) breakthrough curve for the 50% port is shown in Figure 4, compared with the column effluent breakthrough curve. The resin bed volume and EBCT for the 50% port are half those of the whole resin column. Thus, the amount of water treated in number of bed volumes at the 50% port is twice that for the column effluent. Cr(VI) concentration in 50% port samples was mostly below 2 ppb (except two data points in the beginning) until approximately 184,000 BVs when the EBCT was reduced. Then Cr(VI) concentration rose quickly to approximately 7 ppb and fluctuated in the range of 5 to 9 ppb (except one data point) until the end of the pilot test. A total of 397,000 BVs of water was treated by the 50% port. The dramatic increase in Cr(VI) concentration is likely due to the reduced EBCT, considering the slow rate of Cr(VI) removal by WBA resins.

By comparison, Resins A and B were tested in 2012- 2013. The influent Cr(VI) concentrations were mostly between 20 and 30 ppb, except at the end of testing when levels rose to 68 ppb. Resin A effluent showed Cr(VI) leakage starting at 2,300 BVs, in which Cr(VI) concentrations reached up to 10 ppb. However, Cr(VI) levels decreased to 0.025 ppb or less after approximately 30,000 BVs (approximately two months of operation). Subsequently, Cr(VI) remained non-detect (<0.02 ppb) until approximately 88,700 BVs and slowly increased to 1.8 µg/L at approximately 105,200 BVs. Resin B effluent also showed an initial Cr(VI) leakage up to 10 ppb between 4,400 and 51,900 BVs. Subsequently, Cr(VI) concentrations fluctuated between non-detect and 4 ppb until levels rose again at approximately 189,000 BVs. The Cr(VI) concentration was 6 ppb in the end of testing at approximately 200,000 BVs (9 months of operation).

The demonstration-scale test results with PWA7 at the same well site during 2010 - 2012 are shown in Figure 5. The treatment process included two vessels in a lead/lag configuration. When the lead bed was replaced, the lag bed was switched to the lead position. Cr(VI) concentration variations reflected in the resin effluents were mostly due to pH variations during the 2010-2012 period. The influent Cr(VI) concentration at the time was mostly between 25 and 40 ppb. PWA7 resin effluent Cr(VI) went beyond 10 ppb at approximately 200,000 BVs (as represented by the green curve).

Overall, the new WBA resin did not show the Cr(VI) leakage observed for the other two resins A and B. The effect of influent Cr(VI) concentration on chromium removal by the WBA resins is likely minor, based on the test results of a Water Research Foundation study (Najm et al., 2014). Overall, the new WBA resin was tested effective for a greater bed volumes than Resin A, lower and more consistent Cr(VI) effluent concentrations than Resin B and PWA7.

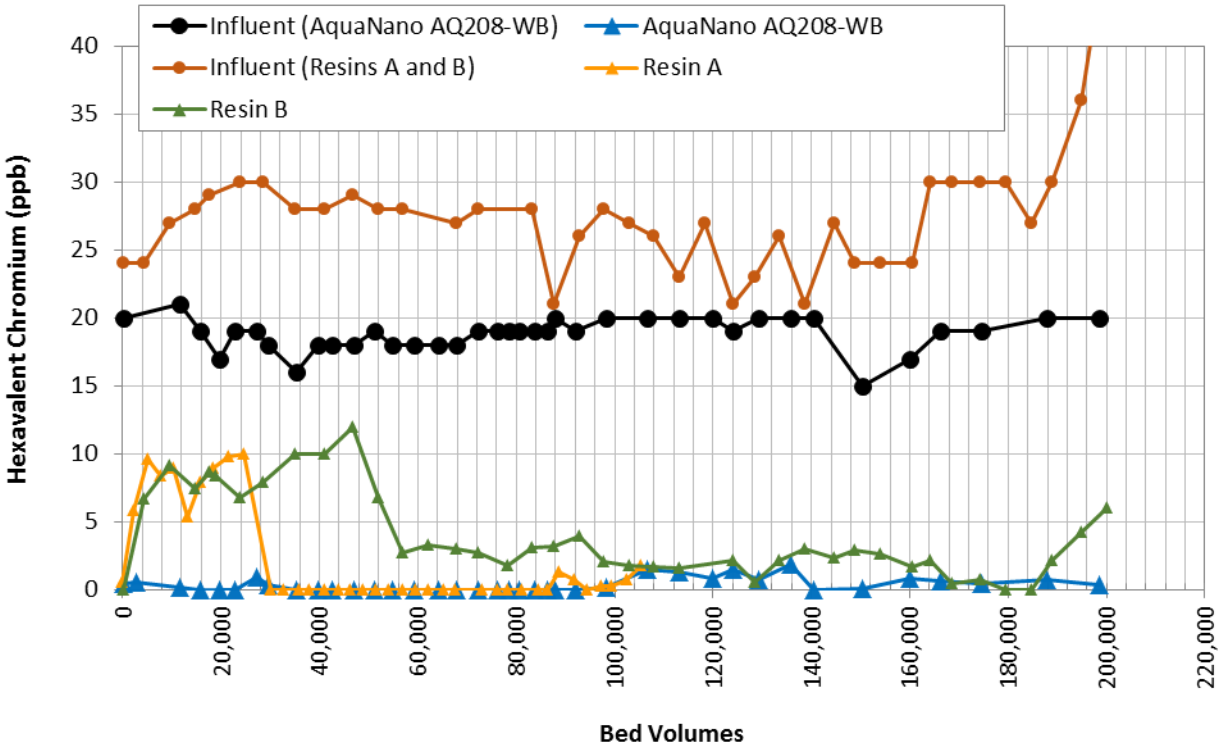

Figure 3. WBA Cr(VI) Breakthrough Curves

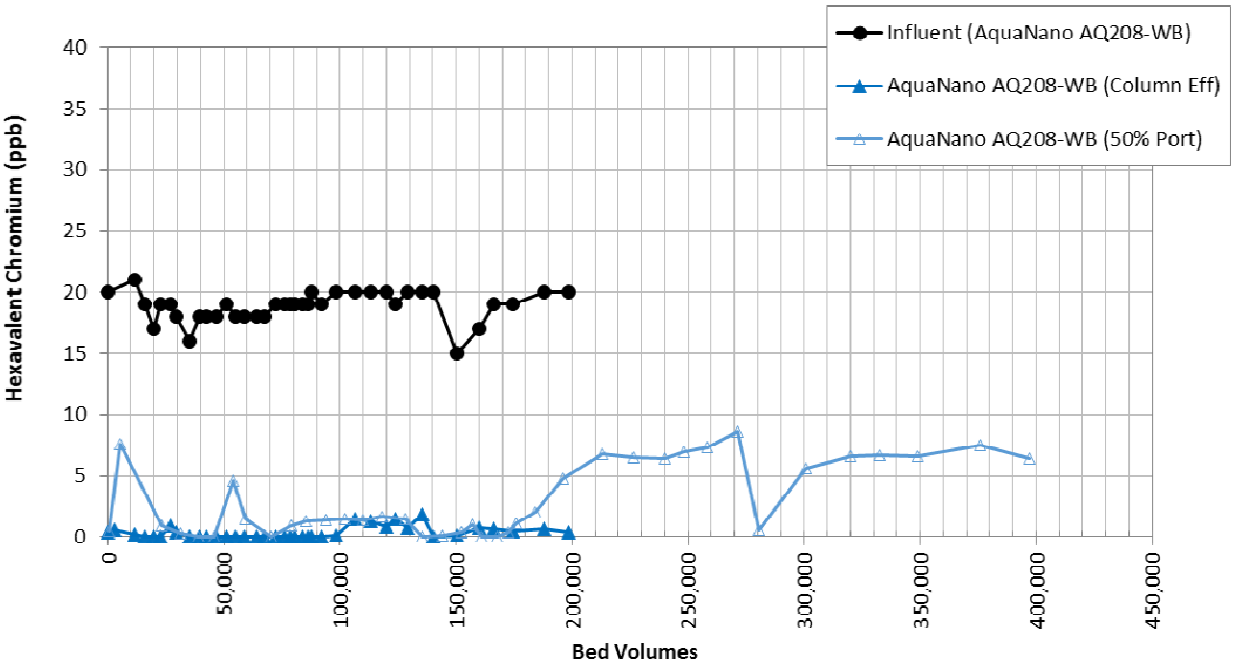

Figure 4. WBA Cr(VI) Breakthrough Curves of 50% Port vs. Column Effluent

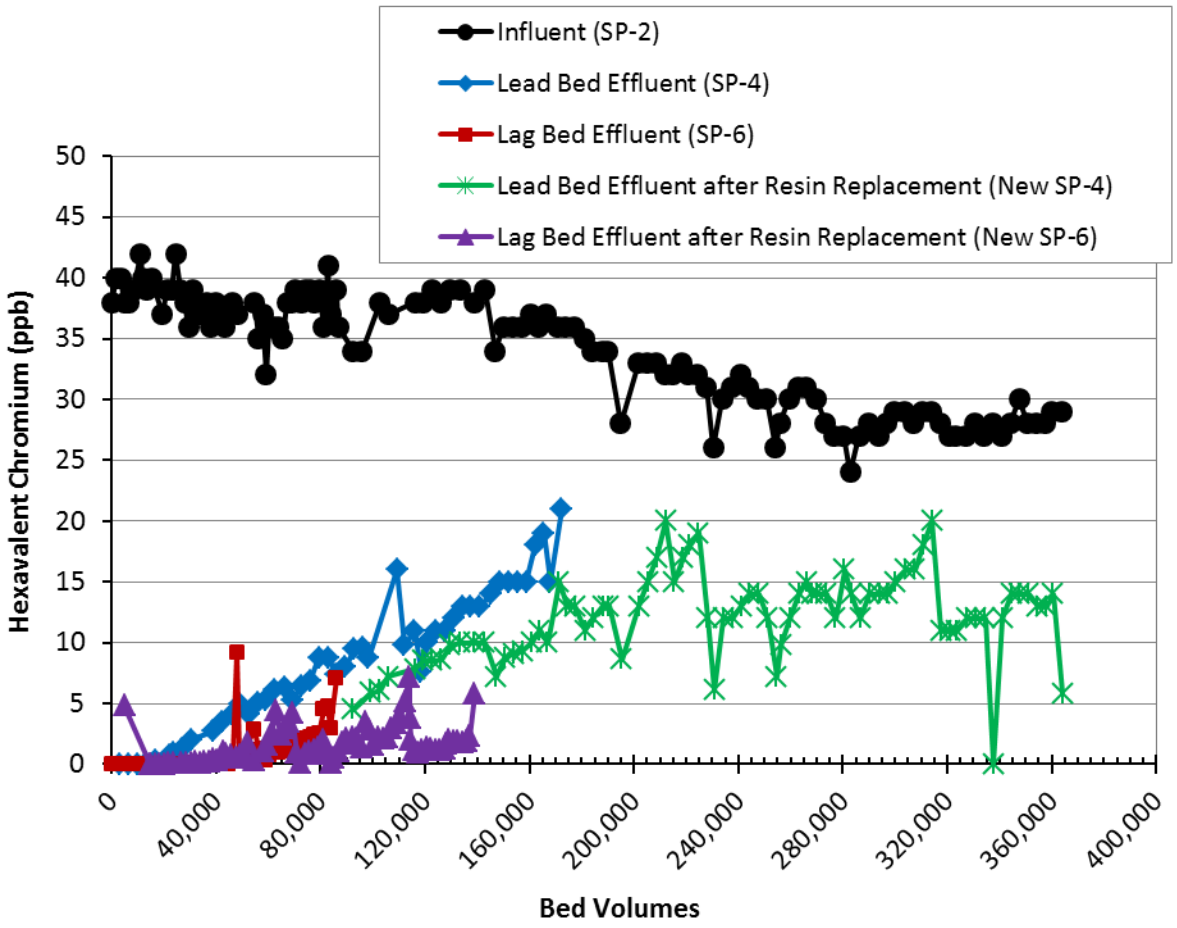

Figure 5. PWA7 Cr(VI) Breakthrough Curves from Demonstration Testing at Glendale
Source: Blute et al. 2014

The total Cr breakthrough curve for the new WBA resin is shown in Figure 6, compared with the two other WBA resins tested in previous studies. Similar trends as Cr(VI) were observed for total Cr. Total Cr in the new WBA resin effluent was mostly <0.2 ppb until 92,000 BVs, when the EBCT was decreased to 1 minute. Afterwards, total Cr levels fluctuated between 0.32 and 2.1 ppb. The final total Cr concentration was 1.2 ppb at approximately 198,600 BVs. The total Cr breakthrough curve for the 50% port is shown in Figure 7. Similar trends as Cr(VI) in Figure 4 are observed. The demonstration-scale test results for PWA7 are shown in Figure 8. Similar to the Cr(VI) results, the new WBA resin did not exhibit the total Cr leakage like the other two WBA resins A and B. Overall, the new WBA resin showed greater total Cr capacity than the other resins, including PWA7.

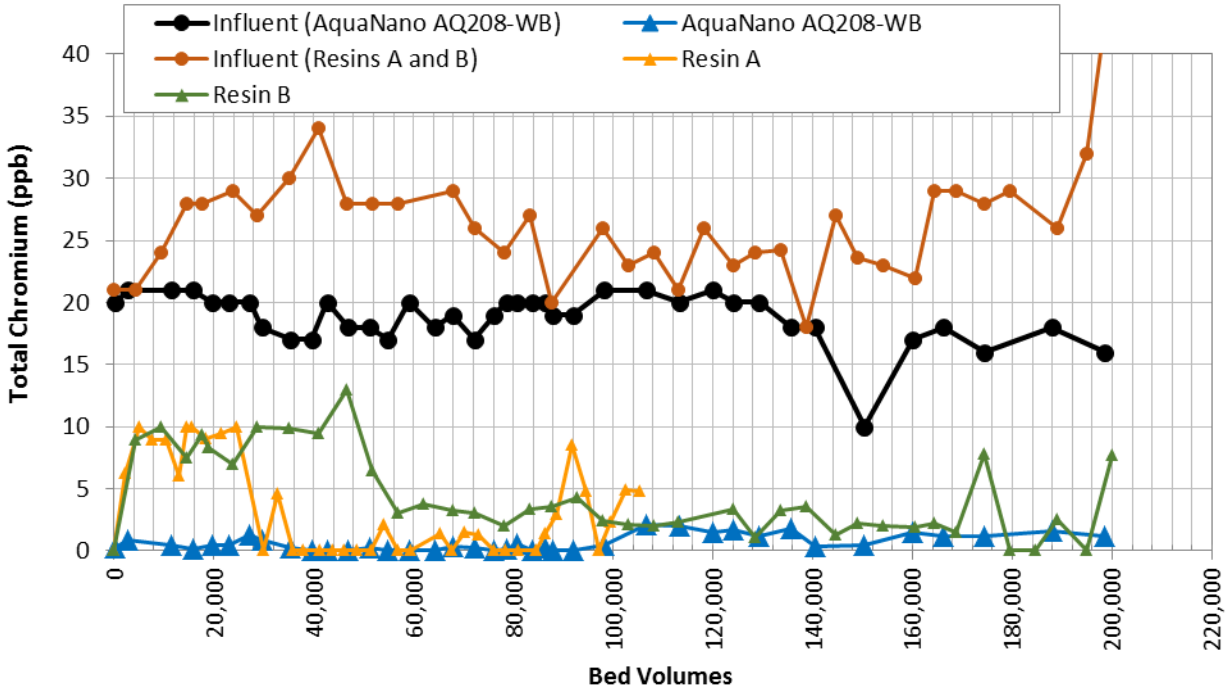

Figure 6. WBA Total Cr Breakthrough Curves

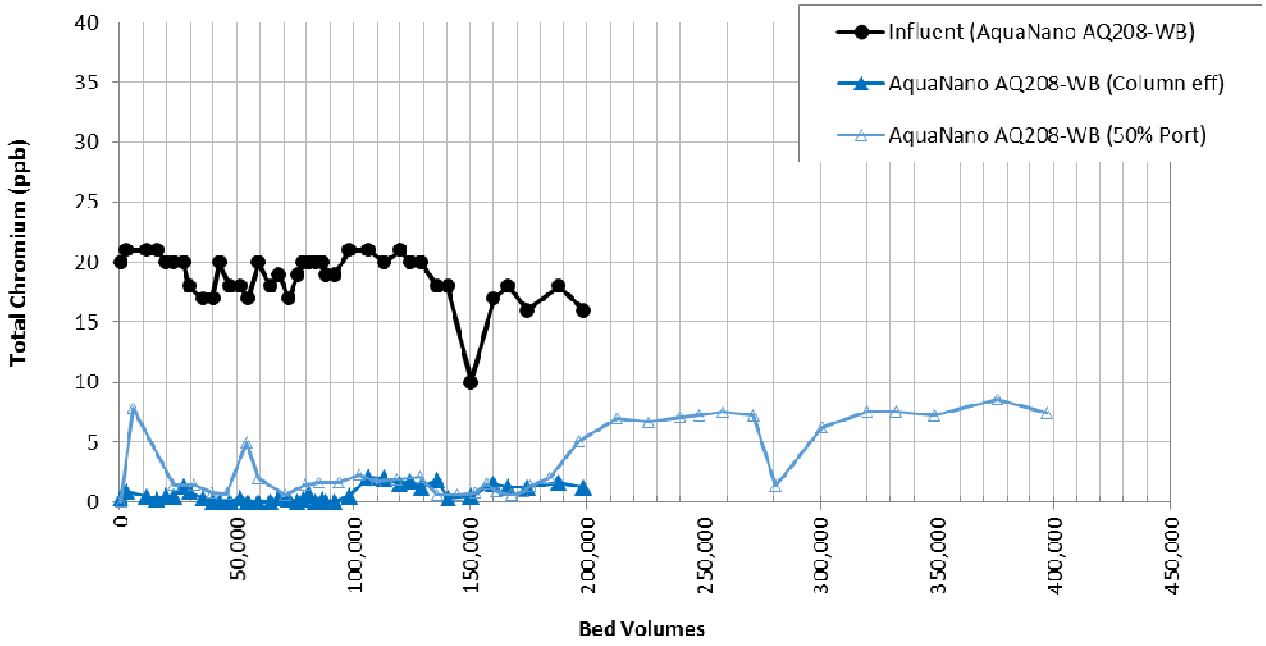

Figure 7. WBA Total Cr Breakthrough Curves of 50% Port vs. Column Effluent

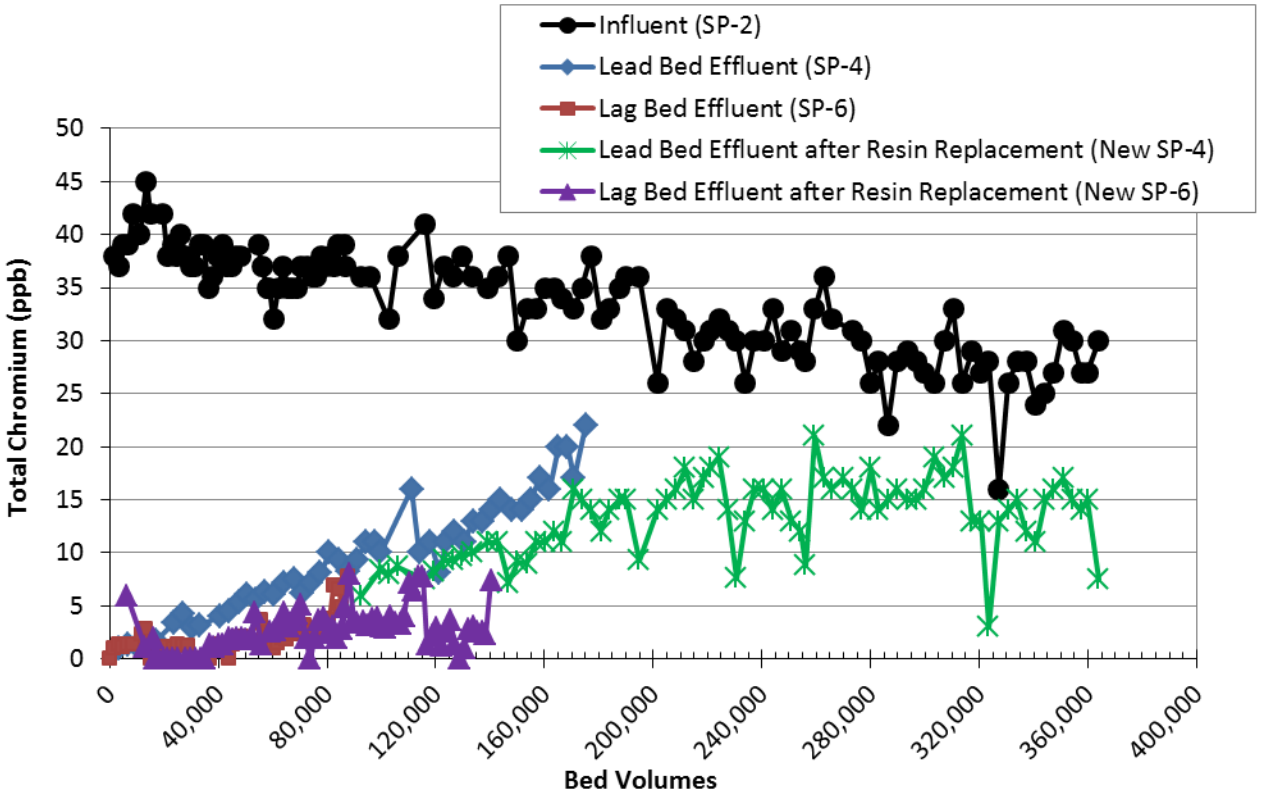

Figure 8. PWA7 Total Cr Breakthrough Curves from Demonstration Testing at Glendale
Source: Blute et al. 2014

Simultaneous Removal of Other Contaminants

Other constituents monitored during the WBA pilot included alkalinity, nitrate, sulfate, uranium, and phosphate. Alkalinity, nitrate, sulfate, and phosphate were non-detect in the startup samples and reached the raw water concentrations in the first monthly samples. Thus, these constituents were initially removed, but broke through the WBA resin relatively fast. Uranium remained non-detect in the WBA resin effluent throughout the 9-month test period, suggesting effective removal by the resin for the duration.

Constituents Leaching

A total of 172 organic compounds were tested at startup, midpoint after a shutdown and restart, and at the end of testing (without a shutdown), including nitrosamines, SVOCs and TICs, VOCs and TICs, ketones/aldehydes. During startup, nitrosamines samples were collected from the raw water and the WBA resin effluent at first flush and after 4 hours of operation. All nitrosamines were non-detect (< 2 ng/L) in the raw water. The only nitrosamine detected in the resin effluent was N-Nitrosodibutylamine or NDBA (2.5 ng/L at first flush), which became non-detect (< 2 ng/L) after 4 hours of operation. NDBA was also detected (5.2 ng/L) at midpoint after a shutdown and restart. No other nitrosamines were detected. NDBA does not have a notification

level in California. N-Nitrosodiethylamine (NDEA), N-Nitrosodimethylamine (NDMA) and N-Nitrosodi-n-propylamine (NDPA) have notification levels of 10 ng/L. Thus, nitrosamines leaching is not a great concern for this new WBA resin.

The majority of the organic compounds tested were non-detect or below levels in the raw water. The constituents detected at concentrations higher than in the raw water are summarized in Table 7. From a regulatory perspective, only 1,3-dichloropropane, carbon tetrachloride, PCE, TCE were detected in the WBA resin effluent above the MCLs. The raw water was known to contain PCE and TCE as well as carbon tetrachloride, cis-1,2-Dichloroethylene, chloroform, and trichlorofluoromethane. Thus, the detection of these compounds in the WBA resin effluent at similar levels as in the raw water was likely due to their presence in the raw water, rather than leaching from the resin. The presence of 1,3-dichloropropane in the WBA resin effluent is unexpected by the resin manufacturer as this compound was neither used in the manufacturing process, nor expected to be formed as a byproduct from potential decomposition. Additional testing is recommended to determine if 1,3-dichloropropane is an issue and if so, attenuation with flushing.

Table 7. Organics with Higher Concentrations than in the Raw Water for WBA

Constituent (unit)	Raw Water	WBA Effluent			Regulatory Limit
		Startup	Midpoint after Shutdown	End of Testing without Shutdown	
1,3-Dichloropropane (µg/L)	< 0.5	7.9	< 0.5	< 0.5	0.5 (MCL)
Acetaldehyde (µg/L)	< 1	1.1	400	< 1	N/A
Butanal (µg/L)	< 1	< 1	4.5	< 1	N/A
Carbon Tetrachloride (µg/L)	0.66 - 1.1	< 0.5	0.59	1.4	0.5 (MCL)
cis-1,2-Dichloroethylene (µg/L)	< 0.5 - 0.68	< 0.5	< 0.5	0.7	6 (MCL)
Chloroform (ug/L)	0.73 – 0.86	< 0.5	0.9	0.94	Note below
Crotonaldehyde (µg/L)	< 1	< 1	3.8	< 1	N/A
Cyclohexanone (µg/L)	< 1	< 1	1.1	< 1	N/A
Glyoxal (µg/L)	< 10	< 10	35	< 10	N/A
NDBA (ng/L)	< 2	2.5 (first flush) < 2 (4 hours)	5.2	< 2	N/A
Propanal (µg/L)	< 1	< 1	1.7	< 1	N/A
Tetrachloroethylene (PCE) (µg/L)	7.6 – 8.8	3.9	7.8	7.9	5 (MCL)
Toluene (µg/L)	< 0.5	17	< 0.5	< 0.5	150 (MCL)
Trichloroethylene (TCE) (µg/L)	19 - 45	8.8	27	50	5 (MCL)
Trichlorofluoromethane (µg/L)	1.0 – 1.3	0.67	0.78	1.3	150 (MCL)

N/A – not applicable, no regulatory limit.
Chloroform is regulated under the MCL of 80 µg/L for total trihalomethanes.
Bolded compounds were detected/existing in the raw water.

Resin Effluent pH during Startup

The WBA resin effluent pH was noted significantly below the raw water pH during startup. Figure 9 shows the field pH tested on Day 1, Day 3 and Day 5 following the start of the field testing. Effluent pH was tested as 3.93 on Day 1 and 5.77 on Day 3, compared to approximately 6 in the resin influent. If applied at full scale, it is recommended to have the fresh resin flushed before installation or blend the fresh resin effluent with other treated water (for example from another ion exchange vessel if possible), to minimize the potential low pH water entering the distribution system.

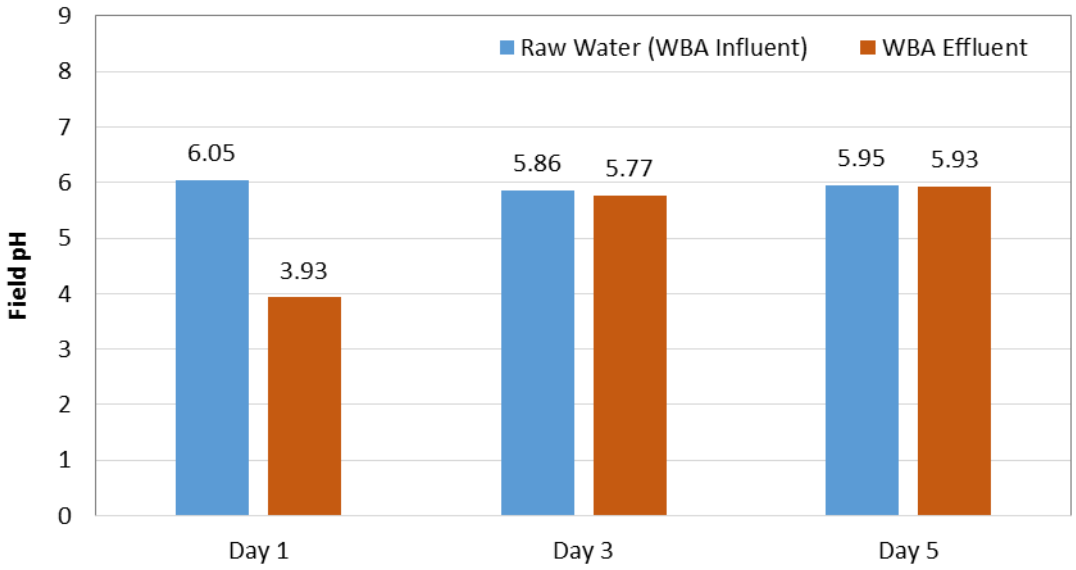

Figure 9. WBA Effluent pH during Startup

Spent Resin Characteristics

Spent WBA resins were characterized using TCLP (metals only), California WET (Total Threshold Limit Concentration or TTLC and Soluable Threshold Limit Concentration or STLC) and uranium. The results are summarized in Table 8. The resin passed the TCLP test, which means it is not a federally classified hazardous waste according to Resource Conservation and Recovery Act (RCRA). The resin also passed the California STLC test but did not pass the TTLC due to chromium concentration above the regulatory limit. Therefore, the spent WBA resin would be classified as a California hazardous waste or non-RCRA hazardous waste, under the conditioned tested in this study. The spent resin would need to be disposed to a non-RCRA hazardous waste landfill.

The regulatory limit for uranium is 0.05% (500 mg/kg) by weight, above which a material is classified as a low level radioactive waste. The uranium concentrations were 1,700 mg/kg (0.17%) based on wet weight. Thus, the spent resin would be classified as radioactive materials and require special handling and disposal. The results are consistent with the water quality sampling showing effective uranium removal from the water. City of Glendale added an absorbent material when disposed PWA7 resin to the Grandview landfill in Idaho in the past, which decreased uranium content below the 0.05% threshold. However, there may be a limit of the amount of absorbent material beyond which the landfill becomes unwilling to accept. It is recommended to check with this and/or other landfills for their acceptable adsorbent material quantity. Another option to avoid generating a low level radioactive waste is to run the WBA resin for a shorter period so that uranium accumulated in the resin is below 0.05% by weight. However, this means a shorter resin life thus more resin replacements. A cost analysis is

recommended for utilities to see if this practice is cost effective based on site-specific water quality and treatment goals.

Table 8. WBA Spent Resin TCLP, CWET and Uranium Results

Metals	TTLC (mg/kg)	TCLP (mg/L)	STLC (mg/L)	TTLC Regulatory Limit (mg/kg)	TCLP Regulatory Limit (mg/L)	STLC Regulatory Limit (mg/L)
Antimony	<2	N/A	<0.52	500	N/A	15
Arsenic	<2	<0.02	<0.52	500	5.0	5.0
Barium	0.95	0.18	<0.13	10,000#	100	100
Beryllium	2.8	N/A	<0.13	75	N/A	0.75
Cadmium	<0.5	0.0078	<0.13	100	1.0	1.0
Chromium	**7,500**	0.15	7.1	2,500	5.0	5^
Cobalt	4.6	<0.005	<0.13	8,000	N/A	80
Copper	770	N/A	2.2	2,500	N/A	25
Lead	12	<0.015	<0.39	1,000	5.0	5.0
Mercury	0.11	<0.0002	<0.0093	20	0.2	0.2
Molybdenum	95	N/A	<0.26	3,500*	N/A	350
Nickel	1.3	N/A	<0.26	2,000	N/A	20
Selenium	<2	<0.02	<0.52	100	1.0	1.0
Silver	0.86	<0.005	<0.13	500	5.0	5
Thallium	<3	N/A	<0.78	700	N/A	7.0
Vanadium	370	N/A	1.4	2,400	N/A	24
Zinc	8.5	<0.02	<0.52	5,000	N/A	250
Uranium	**1,700**	0.064	0.049	N/A	N/A	N/A

N/A – not applicable.
^560 mg/L if passed TCLP test.
#Excluding barium sulfate.
*Excluding molybdenum disulfide.
Uranium results are based on wet weight.
Bolded results are above the regulatory limit.

SBA Results

The SBA resin was tested with two EBCTs, 2 minutes and 4 minutes. The SBA resin was evaluated for removal of Cr(VI) and total Cr, removal of other contaminants, constituents leaching at startup and after a shutdown, spent resin characteristics for disposal.

Chromium Removal

Cr(VI) breakthrough curve for the new SBA resin is shown in Figure 10, compared with two other SBA resins tested in previous studies. Cr(VI) concentration in the 2-min EBCT column effluent rose from <0.02 ppb during the startup to 12 ppb (below the influent concentration) at approximately 18,200 BVs. Then Cr(VI) concentration remained relatively stable and rose very slowly to 16 ppb at approximately 128,600 BVs, which it is still below the influent concentration. The 4-min EBCT column showed similar Cr(VI) breakthrough as the 2-min EBCT column. Thus, the longer EBCT did not demonstrate a significant benefit for Cr(VI) removal. The resin effluent Cr(VI) level reached 8 ppb between approximately 12,000 to 14,000 BVs, which is about 17 to 19 operational days with a 2-minute EBCT.

In comparison, Resins C and D were tested with the same well water during 2012 – 2013, when the influent Cr(VI) concentration was 27 ppb on average. Resin C reached full Cr(VI) breakthrough at approximately 6,500 BVs. Resin D effluent Cr(VI) reached 22 ppb at approximately 4,300 BVs ultimately increasing to 27 ppb. The resin life for Resins C and D to reach 8 ppb Cr(VI) is approximately 4 to 5 days with a 2-minute EBCT. Overall, the new SBA resin showed a significantly improved Cr(VI) capacity compared to the resins tested previously.

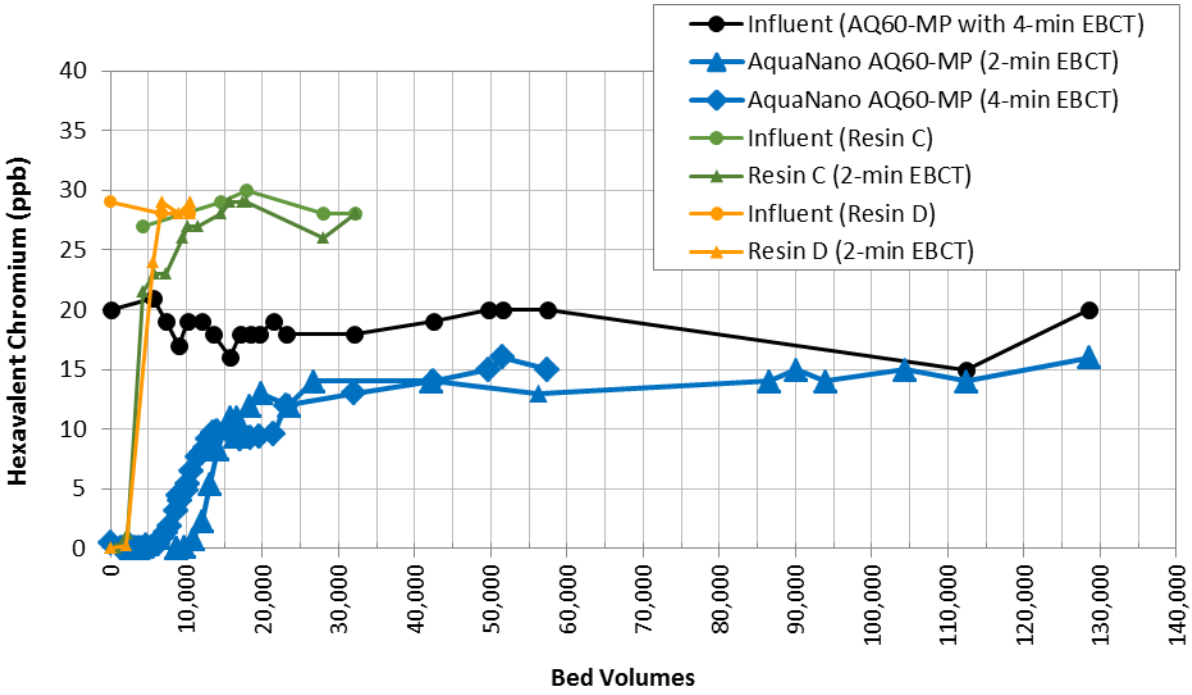

Figure 10. SBA Cr(VI) Breakthrough Curves

Total Cr results for the new SBA resin are shown in Figure 11, compared to Resins C and D tested previously at Glendale. Similar trends are observed as Cr(VI) results, as Cr(VI) is the dominant species of chromium in the water. Total Cr in the 2-min EBCT resin effluent reached 8.3 ppb at approximately 14,100 BVs and stabilized between 13 to 16 ppb during the rest of the test period. The longer EBCT (4 minutes) did not demonstrate a significant improvement for total Cr removal. Overall, the new SBA resin showed significantly improved total Cr removal capacity compared to Resins C and D.

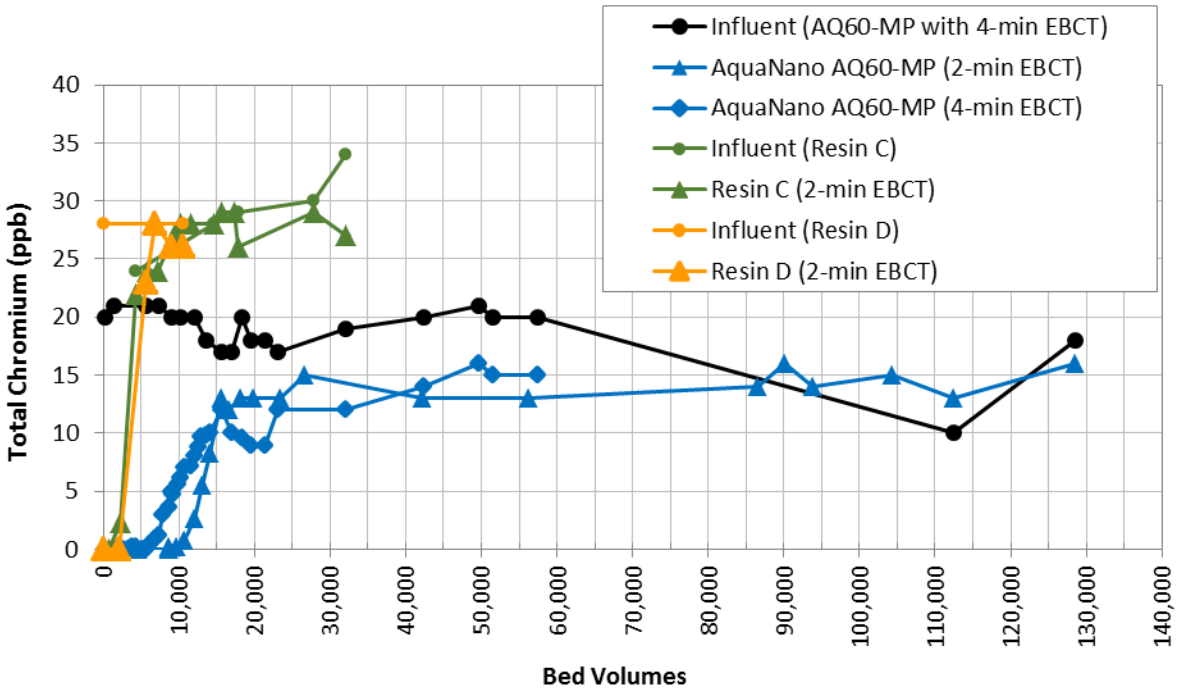

Figure 11. SBA Total Cr Breakthrough Curves

Simultaneous Removal of Other Contaminants

Other constituents monitored during the SBA pilot included alkalinity, nitrate, sulfate, uranium, and phosphate. Alkalinity, nitrate, sulfate, and phosphate were non-detect in the startup samples and reached the raw water concentrations in the first three-times-a-week samples (three days after startup). Thus, these constituents were initially removed, however, broke through the SBA resin relatively fast. Uranium remained non-detect in the SBA resin effluent until the end of the testing (approximately 163,000 BVs), suggesting effective removal by the resin.

Constituents Leaching

Similar as WBA, the SBA resin with 2-minute EBCT was tested at startup and midpoint after a shutdown and restart for organic compounds, including nitrosamines, SVOCs and TICs, VOCs

and TICs, ketones/aldehydes. The majority were non-detect or below raw water levels. The constituents detected at concentrations higher than in the raw water are summarized in Table 9. NDBA was detected in the first flush sample (2.7 ng/L) and became non-detect after 4 hours of operation. NDMA was detected as 4.8 ng/L in the first flush sample, which was below the 10 ng/L notification level in California. NDMA was non-detect (< 2 ng/L) in the 4 hour sample. Thus, nitrosamines are not a great concern for this SBA resin. Four other organic compounds were detected at startup or midpoint after a shutdown and restart, including 2-butanone, propanal, glyoxal and methyl glyoxal. None of them are regulated at federal level or in California.

Table 9. Organics with Higher Concentrations than in the Raw Water for SBA

Constituent (unit)	Raw Water	SBA Effluent (Lower EBCT)		Regulatory Limit
		Startup	Midpoint after Shutdown	
NDBA (ng/L)	< 2	2.7 (first flush) <2 (4 hours)	< 2	N/A
NDMA (ng/L)	< 2	4.8 (first flush) <2 (4 hours)	< 2	10 (CA Notification Level)
2-Butanone (MEK) (µg/L)	< 5	46	< 5	N/A
Propanal (µg/L)	< 1	< 1	14	N/A
Glyoxal (µg/L)	< 10	< 10	11	N/A
Methyl glyoxal (µg/L)	< 10	< 10	14	N/A

N/A – not applicable, no regulatory limit.

Resin Effluent pH during Startup

The SBA resin effluent pH was noted significantly below the raw water pH during startup. Figure 12 shows the field pH tested on Day 1, Day 3 and Day 5 following the start of the field testing. The results suggest resin effluent pH returned back to the raw water level on Day 3. If applied at full scale, it is recommended to have the fresh resin flushed before installation or blend the fresh resin effluent with other treated water (for example from another ion exchange vessel if possible), to minimize the potential low pH water entering the distribution system.

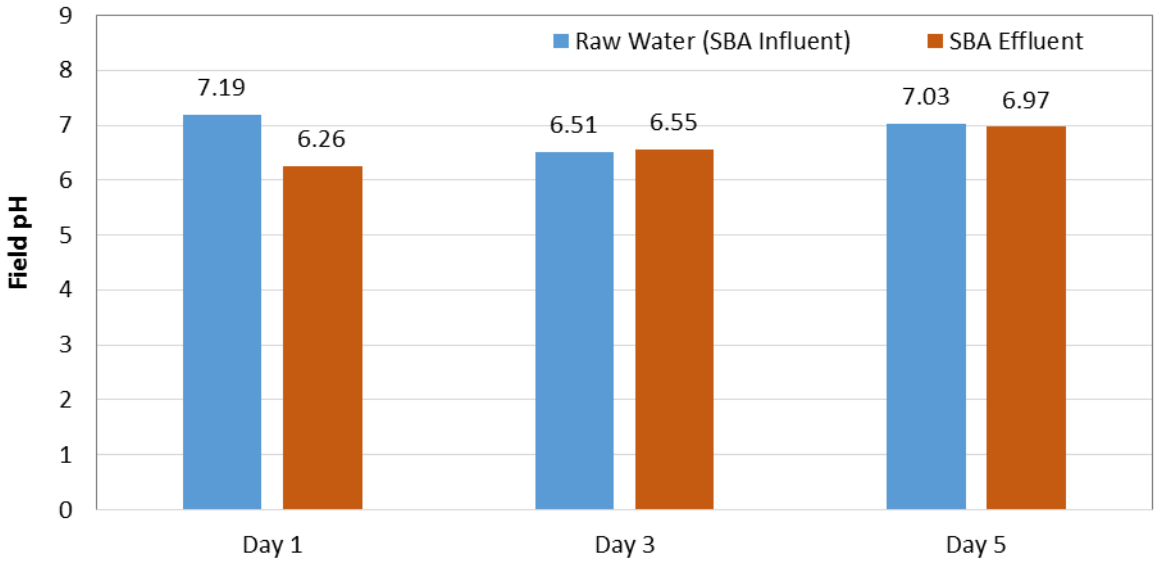

Figure 12. SBA Effluent pH during Startup

Spent Resin Characteristics

Spent SBA resins with 2-minute and 4-minute EBCT were characterized using TCLP (metals only), CWET (TTLC and STLC) and uranium. The results are summarized in Table 10. Both resins passed the TCLP test, indicating the SBA resin is likely not a federally classified hazardous waste. Both resins passed the CWET TTLC and STLC tests. Therefore, the spent resin is not a hazardous waste in California.

Uranium concentrations in the SBA spent resin are 0.03% and 0.05% by weight (based on wet weight) for 2-minute and 4-minute EBCT, respectively. This is consistent with the effective uranium removal in the water quality samples. Materials with uranium above 0.05% by weight is classified as a radioactive material, which requires special handling and disposal as discussed in the WBA spent resin section. Uranium concentration in spent resin is affected by the SBA resin life and uranium in the raw water. When applied at full scale, the SBA resin may not be operated for such a long period as tested in this study, considering utilities typically want to keep treated Cr(VI) below the MCL of 10 ppb. In this case, the uranium concentration in the spent resin might be kept below 0.05% by weight. Mass balance can be used as a simple tool to estimate uranium content of the spent resin for utilities.

Table 10. SBA Spent Resin TCLP, CWET and Uranium Results

Metals	SBA (Lower EBCT)			SBA (Higher EBCT)			TTLC Regulatory Limit (mg/kg)	TCLP Regulatory Limit (mg/L)	STLC Regulatory Limit (mg/L)
	TTLC (mg/kg)	TCLP (mg/L)	STLC (mg/L)	TTLC (mg/kg)	TCLP (mg/L)	STLC (mg/L)			
Antimony	<2	N/A	<0.52	<1.9	N/A	<0.52	500	N/A	15
Arsenic	<2	<0.5	<0.52	<1.9	<0.02	<0.52	500	5.0	5.0
Barium	7.8	0.04	0.7	6.2	<0.005	0.71	10,000#	100	100
Beryllium	2.7	N/A	<0.13	2.6	N/A	<0.13	75	N/A	0.75
Cadmium	7.8	<0.005	<0.13	<0.49	<0.005	<0.13	100	1.0	1.0
Chromium	640	0.037	2.2	570	<0.015	1.7	2,500	5.0	5^
Cobalt	1.2	<0.005	<0.13	1.2	<0.005	<0.13	8,000	N/A	80
Copper	450	N/A	2.3	310	N/A	1.8	2,500	N/A	25
Lead	3.3	<0.015	<0.39	2.4	<0.015	<0.39	1,000	5.0	5.0
Mercury	<0.099	<0.0002	<0.0083	<0.097	<0.0002	<0.0083	20	0.2	0.2
Molybdenum	10	N/A	<0.26	11	N/A	<0.26	3,500*	N/A	350
Nickel	<0.98	N/A	<0.26	<0.97	N/A	<0.26	2,000	N/A	20
Selenium	2.0	0.009	<0.52	1.7	<0.02	<0.52	100	1.0	1.0
Silver	<0.49	<0.005	<0.13	<0.49	<0.005	<0.13	500	5.0	5
Thallium	<2.9	N/A	<0.78	<2.9	N/A	<0.78	700	N/A	7.0
Vanadium	370	N/A	0.62	370	N/A	0.53	2,400	N/A	24
Zinc	6.5	0.014	<0.52	5.1	<0.02	<0.52	5,000	N/A	250
Uranium	330	0.015	<0.013	**540**	0.01	<0.013	N/A	N/A	N/A

^560 mg/L if passed TCLP test
#Excluding barium sulfate.
*Excluding molybdenum disulfide.
Bolded results are above the TTLC regulatory limit.
Uranium based on wet weight.

V. Summary and Conclusions

Results of this study showed the new WBA resin (AQ208-WB) had a high Cr(VI) capacity without the initial Cr(VI) leakage observed with the other two resins (A and B) tested at Glendale in 2012-2013. The influent Cr(VI) concentration was slightly lower than tested for Resins A and B, although it was the same water source. The impact of influent Cr(VI) level on resin life has not been quantified. However, a Water Research Foundation study suggests the impact of Cr(VI) levels on WBA resin life is likely minor (Najm et al., 2014). Findings from testing of AquaNano resin indicate that the new WBA resin performs at least as well if not better in terms of Cr(VI) capacity, and does not initially leach Cr(VI).

The new WBA resin removed alkalinity, nitrate, sulfate and phosphate, although fast breakthrough occurred (less than one month – by the second sample collected). Uranium was effectively removed throughout the 9-month test period. Resin effluent pH was noted to be lower

on the first day of testing, which returned to the influent pH in less than three days. Nitrosamines, VOCs, SVOCs, aldehydes and ketones leaching was either not detected or at levels similar to the raw water (except 1,3-dichloropropane). Formaldehyde was not detected. The spent WBA resin from this study was classified as a non-RCRA hazardous waste (hazardous in California) due to chromium concentration above the TTLC regulatory limit. In addition, uranium was also above the regulatory limit for low level radioactive material. At full-scale, the uranium concentration in the spent resin could be controlled by reducing the resin life, which would result in more frequent resin change-outs. Adding absorbent materials to spent resin during disposal to minimize water may also have an impact on uranium content. Landfills should be consulted for specific requirements and limits, including the amount of absorbent materials permissible.

The new SBA resin (AQ60-MP) was tested as single pass without regeneration. The test water contained relatively high sulfate (110 – 120 mg/L), which is expected to negatively impact SBA resin life. The results show significantly greater Cr(VI) capacity of the AquaNano resin compared to other SBA resins tested at Glendale during 2012-2013. Resin effluent Cr(VI) reached 8 ppb at approximately 12,000 to 14,000 BVs (two columns), which is about 17 to 19 operational days with a 2-minute EBCT. By comparison, the other SBA resins had an operational life of 4 to 5 days when tested with the same water. A greater EBCT (4 minutes) did not improve Cr(VI) capacity or resin life for the new SBA resin.

Simultaneous removal of other constituents, including alkalinity nitrate, sulfate, and phosphate were observed but with faster breakthrough than Cr(VI) (less than three days – by the second sample collected). Uranium was effectively removed throughout the 9-month test period. Resin effluent pH was reduced to 6.3 (compared to the influent pH of 7.2) on the first day of testing, and then returned back to the influent pH level in less than three days. Constituents leaching testing revealed the majority were either not detected, or at lower levels than in the raw water. Four organic compounds were detected at startup or midpoint after a shutdown and restart, including 2-butanone (46 µg/L), propanal (14 µg/L), glyoxal (11 µg/L), and methyl glyoxal (14 µg/L). None of these constituents have a regulatory limit at federal level or in California. Spent SBA resin are likely characterized as a non-hazardous waste by federal and California standards. The uranium concentration of the spent resin with 2-minute EBCT was below the regulatory limit for the conditions tested, although it is still considered TENORM. Disposal options depend on landfill-specific limits. The uranium concentration in the spent resin is affected by raw water uranium and resin operational life. Thus, it may increase or decrease for other water agencies. Resin regeneration was not tested in this study, and is recommended to evaluate the resin performance with regeneration.

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