

A Standardized Test Protocol for Evaluation of Scale Reduction Technologies

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Abstract

Precipitation of calcium carbonate (i.e., scaling) can occur in both traditional tank (electric and gas) and “green” tankless hot water systems that have implications for public health, water and energy sustainability, infrastructure damage, and consumer esthetics. There are many scale reduction devices and technologies that aim to reduce or eliminate such problems, and several standardized methods have been proposed to research their performance with scientific rigor. All of the existing approaches were inherently nonreproducible or could not quantify important aspects of scale deposition, including quantity, location, and deposit durability. Here we develop and vet a Standardized Scaling Test Protocol that overcomes many of these deficiencies, using a laboratory-scale model premise plumbing system and a synthesized synthetic scaling water that could be reproduced in any laboratory. This approach produced 25.1 g of calcium carbonate scaling (95% confidence interval of 20.3–29.8 g, $n=3$) in ~ 5 days. Illustrative scale reduction for a range of representative technologies, including cation exchange, electrochemical deionization, magnetism, electric field generator, media-induced precipitation, phosphate sacrificial media, and citric acid sacrificial media, ranged from 0% to 100% using the standardized protocol. The general approach was also applied to suitable local natural water with high scaling potential, and similar capabilities were observed.

Keywords: calcium carbonate; scale reduction; water heater

Introduction

Calcium carbonate scaling problems within the premise plumbing system

WHILE CALCIUM CARBONATE is traditionally studied for its perceived role in corrosion control (Lytle and Snoeyink, 2002; Sarin *et al.*, 2004), issues with calcium carbonate precipitation in premise plumbing systems are becoming an increasing concern. Scaling in potable water systems is a serious problem affecting both traditional tank water heaters (gas and electric), especially “green” tankless (on-demand) water heaters (Richards *et al.*, 2016). Scaling problems can reduce energy efficiency, recovery times, bacterial regrowth potential, and the lifetime of water

heaters, and it is an important emerging concern in design of building water systems at the cutting edge of the energy–water–public health nexus (Brazeau and Edwards, 2011; Fox and Abbaszadegan, 2013). Scaling can also create many other problems (Table 1) for treatment plants, water main distribution systems, premise plumbing, appliances, and even fixtures, including head loss, flow reduction, clogged pipes, and erosion corrosion damage (Roy and Edwards, 2015). Scale creates unsightly deposits on coffee pots, dish washers, and shower doors. Hard water scales may also exacerbate rashes and growth of opportunistic bacteria (Fox and Abbaszadegan, 2013; Flohr and Mann, 2014).

The scaling propensity of water can vary with temperature and chemistry changes as it passes through the distribution system. For example, in most buildings the scaling potential is maximized at the surface of the water heating elements, and decreases somewhat downstream from this point due to the cooling and softening of the water due to precipitation. The kinetics of precipitation is also controlled by water chemistry and temperature (Richards *et al.*, 2016).

Available approaches for scale reduction

A range of scale reduction technologies are available to reduce problems with scaling, some of which can be applied

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TABLE 1. SUMMARY OF PROBLEMS DUE TO CALCIUM CARBONATE SCALING AT DIFFERENT POINTS OF THE POTABLE WATER SYSTEM

<i>Control points</i>	<i>Problems due to calcium carbonate (CaCO₃) scaling</i>
Water main distribution system	Head loss, clogged pipes
Hot water system plumbing	Energy loss, flow reduction
Gas water heater	Energy loss, flow reduction
Electric water heater	Energy loss, profound flow reduction, clogging
On-demand water heater	Erosion corrosion damage, clogged pipes
Downstream plumbing	Malfunctions from erosion corrosion and clogging
Valves	CaCO ₃ scaling of fixture
Consumers	CaCO ₃ residue on glassware
Coffee machine	CaCO ₃ scaling leaves spots/deposits on shower door
Dish washer	CaCO ₃ scaling and soap creates scum in bathtub
Shower door	High soap demand, soap scum, and scaly skin
Bathtub	CaCO ₃ scaling leaves spots on the surface of any equipment that contacts water
Soap demand	Hard water scales may exacerbate rashes or growth of opportunistic bacteria
Bathing surfaces that contact water	
Human health	

at a centralized drinking water treatment plant, and others that are commercially available for application in buildings. The three general approaches used for scale reduction include hardness (i.e., Ca⁺² and Mg⁺²) removal, scaling inhibitors, and methods that might alter precipitate crystal morphology or the location of precipitation.

During hardness removal (i.e., cation exchange water softening and electrochemical deionization), calcium ions are removed from the water to reduce or eliminate the driving force for calcium carbonate scaling. While these devices are effective, they are expensive, and there is a net addition of salt to the water discharged from the homes using softeners that increases the salinity of receiving waters.

Inhibitors (e.g., orthophosphate and polyphosphate) are commonly used in water distribution systems for sequestration, antiscaling, and corrosion control. Phosphates are able to inhibit calcium carbonate precipitation by absorbing onto calcite crystal growth sites, which blocks further growth (Lin and Singer, 2005, 2006). After the Lead and Copper Rule was implemented by the United States Environmental Protection Agency in 1991, an increased number of utilities began using inhibitors (mainly phosphates, at doses up to 3 mg/L as PO₄) to control levels of lead and copper in drinking water (McNeill and Edwards, 2002). It is estimated that >50% of the larger utilities in the United States are presently using phosphate corrosion inhibitors that would also have some influence on scaling (Edwards and McNeill, 2002; Arnold *et al.*, 2020). Lin and Singer found that total phosphate concentrations >10⁻⁸ M (equating to ~0.001 mg/L PO₄) could inhibit calcium carbonate precipitation in supersaturated solutions ($\Omega=5.2$) at temperatures up to 45°C (Lin and Singer, 2005).

Methods that claim to alter the precipitate crystal morphology and/or location of precipitation include media-induced precipitation, electric field generation, and magnetic water treatment. Media-induced precipitation devices are thought to create precipitates that are either removed in a filter or which stay suspended in the water. These devices do not prevent precipitation, but seek to control where and when the precipitates form, thereby avoiding adverse consequences of uncontrolled scale deposits that adhere to surfaces. In electromagnetic devices, hard water is passed through a

magnetic field, and there are some reports that scaling is altered or reduced through a variety of proposed mechanisms. Evidence suggests that under certain conditions these devices can reduce the amount of scale formed, encourage precipitate formation in the water rather than on surfaces, produce less tenacious scale by changing the crystal morphology of scales, remove existing scale, and/or prevent scaling for an extended period of time after treatment (Yang, 2005).

A critical review of current industry standards on scale evaluation

There are many scale reduction devices whose performance will vary from water to water, or as a function of flow rate, temperature, and other factors—several prior efforts have been made to develop standardized test protocols to serve as a basis for research and performance verification. Our evaluation of these previous efforts revealed certain limitations (Table 2) in terms of their reproducibility, and/or their ability to measure key dimensions of scale reduction, including quantity, location, and durability of the deposit.

We calculated the saturation ratio for calcite (Equation 1) for each synthetic test water used in the current standardized test protocols to illustrate the driving force for scaling of each test water.

$$\Omega_{\text{calcite}} = \frac{\{Ca^{2+}\}\{CO_3^{2-}\}}{K_{sp, \text{calcite}}} \quad (1)$$

If the saturation ratio is equal to 1, the solution is at equilibrium with calcite, and no net dissolution or precipitation can occur. If the saturation ratio is >1, the solution is supersaturated, and calcite can precipitate making the test water unstable.

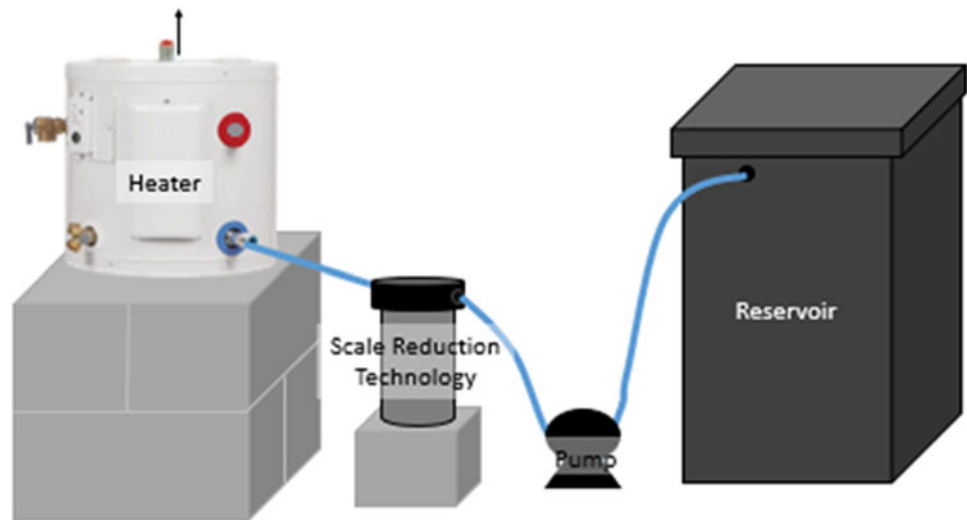
To advance standardized testing approaches and protocols, the Water Quality Research Foundation (WQRF) funded this research to address some limitations of the prior methods through development of a Standardized Scaling Test Protocol (SSTP). The goal was to create a

TABLE 2. EVALUATION OF CURRENT INDUSTRY STANDARDS FOR EVALUATING SCALE REDUCTION TECHNOLOGIES

Tested devices	Scale quantification			Water chemistry		Potential for CaCO ₃ to precipitate before scale reduction device? ^a	
	Key measure of scaling	Location (heating element, water heater, downstream plumbing)	Type of scale (total, bulk water, loose/adherent surface scale)	Temperature [°C]	pH		Total hardness [mg/L CaCO ₃]
NSF/ANSI 44 (NSF/ANSI, 2015)	Cation exchange water softeners	Reduction of total hardness in effluent compared with influent	No scale quantification	T _{in} = 18 ± 5	7.5 ± 0.5	342 ± 34.2	Yes
WQA/ASPE 1201 (WQA/ASPE, 2015)	Electrochemical antiscalcing devices	Reduction of total hardness in effluent compared with influent	No scale quantification	T _{in} = 18 ± 2	7.8 ± 0.3	290.7–393.3	Yes
IAPMO Z601 (IAPMO, 2016)	Any scale reduction device	Loss of hardness in apparatus, and the sum of dissolved adherent and loose scales in the entire apparatus	Water heater, downstream plumbing, fixture/appliance	T _{in} = NA ΔT ≥ 50 T _{out} = 65.5	8.0 ± 0.5	450 ± 50 Ca: 75–85% Mg: 15–25%	Yes
IAPMO IGC – 91 (IAPMO, 2019)	Electrical or high-density magnetic scale reduction devices	Measured voltage, pressure loss, and oxidation reduction potential	No scale quantification			Minimum total hardness of 256.5 mg/L as CaCO ₃	
IAPMO IGC– 335 (IAPMO, 2019)	Any scale reduction device	Amount of scale was determined by the difference in weight of the heater sheath after the experiment and after cleaning it in 4 M HCl.	Heating element only	T _{in} = NA T _{out} = 82		The challenge water used for this test is dictated by the manufacturer and could vary widely from test to test, thus hindering the reproducibility of this test	
DVGW Standard W 512 (AWWA, 1996)	Any water conditioning device	Total mass of scale recovered from the heater	All scale within heater is recovered (walls and heating coils)	T _{in} = NA T _{out} = 80 ± 3		Minimum total hardness of 3.5 mol/m ³ (with <25% by mole, magnesium content) Minimum calcium carbonate precipitation potential of 30 mg/L CaCO ₃ (calculated at 15°C)	

^aMINEQL⁺ used to determine precipitation potential of water in revisor (before scale reduction device).
DVGW: Deutsche Vereinigung des Gas- IAPMO, International Association of Plumbing and Mechanical Officials; IGC, IAPMO Guide Criteria; NSF/ANSI, National Sanitation Foundation/American National Standards Institute; WQA/ASPE, Water Quality Association/American Society of Plumbing Engineers.

FIG. 1. SSTP system. Synthetic water was well mixed and stored in an insulated 200-gallon polyethylene tank. Water was peristaltically pumped to a 6-gallon electric tank-type water heater (A.O. Smith EJC-6). Water was heated to 65.5°C where it became supersaturated with calcium carbonate. The water then flowed through a 15.24 m long stainless-steel tubing coil (5.33 mm inner diameter) enclosed in an insulated box (Fig. 3), used to simulate downstream plumbing. SSTP, Standardized Scaling Test Protocol.



protocol that could be reproduced in any laboratory for comparable results in one standardized water, and that could also be modified to evaluate the performance of any scale reduction technology in any water. A key factor in ensuring reproducibility of the protocol was development of a stable standardized influent water, and a means of creating a driving force for precipitation as would naturally occur in hot water premise plumbing, without having a supersaturated influent water that could create variable feedwater chemistry.

Materials and Methods

Operational parameters

The test water was well mixed and stored in an insulated container. Water was pumped from the reservoir to each scale reduction device (using a peristaltic pump outside the reservoir), and then to a 6-gallon electric tank-type water heater

(Fig. 1). A heating element with a watt density of ~ 220 WPSI (representing what is typical for the residential U.S. water heater market) was used for all experiments.

The water was heated to 65.5°C, at which point it became supersaturated with calcium carbonate, and then flowed through a 15.24 m long stainless-steel tubing coil (5.33 mm inner diameter) to simulate downstream plumbing (Fig. 2).

Optimization of any standardized laboratory test protocol involves compromise, in this case to achieve a maximum amount of scale formation under relatively reasonable flow conditions, while also minimizing the time of testing and the volume of water that is used. A representative flow velocity of 0.3 m/s through the coil tubing was selected, with a semicontinuous flow cycle of 10 s on; 39 s off, requiring a volume of 151 L water per 24 h. The velocity of 0.3 m/s is at the lower end of a normal velocity range, and it was selected because it was realistic while also minimizing the required volume of test water (Roy *et al.*, 2018). The calculated Reynolds number (3690) was less than the Critical Reynolds

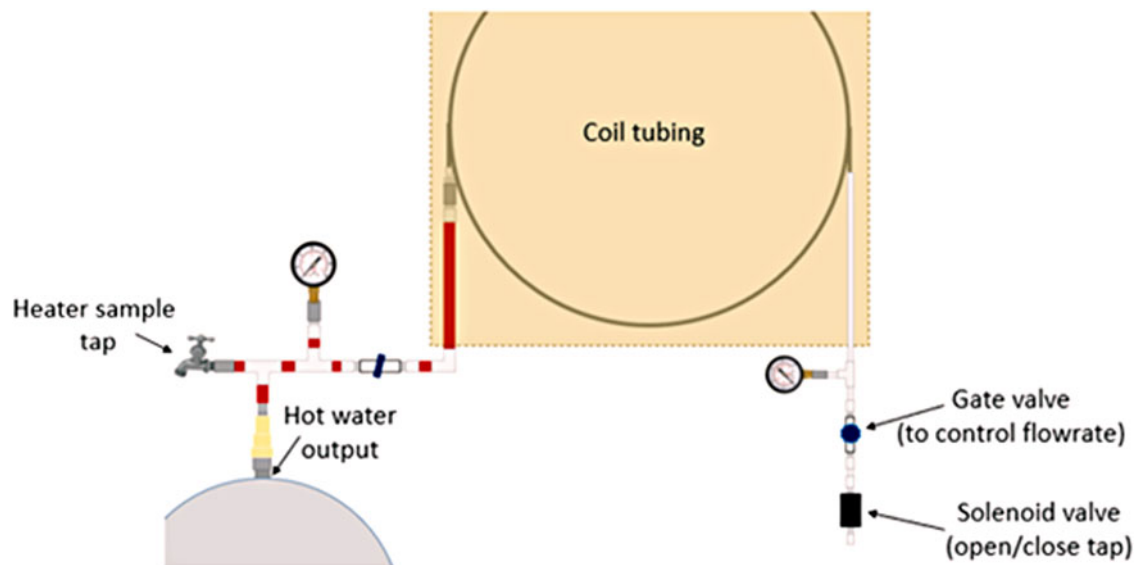


FIG. 2. Heater Output and Downstream Plumbing. The gate valve was used to control the flow rate, and the solenoid valve was used to control the cycle of 10 s on; 39 s off for 5 days (120 h).

number (4620), and the flow is considered laminar (Supplementary Data page 1, Table S1). The 10 s on and off cycle used is $\sim 1/3$ of the median flush duration of 35 s for residential hot water use (DeOreo *et al.*, 2016).

Daily sampling

Filtered and unfiltered samples were taken daily from the cold-water influent to measure pH, temperature, calcium and phosphate (Supplementary Tables S6–S18 and Supplementary Tables S54–S59) to confirm the water was stable throughout the experiment. The flow rate and pressure were measured daily (Supplementary Tables S19–S53) and adjusted by changing the pump speed, and opening or closing the gate valve to achieve a flow rate of 6.8 ± 0.5 mL/s (0.11 ± 0.008 gpm). The pressure within the system was always maintained between 10 and 50 psi to prevent degassing of the test water.

Scale recovery methods

The location and amount of scale were characterized at the end of each experiment. Locations included the heater element, the water heater, and the downstream coil tubing. Two methods were attempted to recover and quantify the scale within the water heater (Supplementary Data). Method 1 used nitric acid to dissolve scale, adapted from International Association of Plumbing and Mechanical Officials (IAPMO)/American National Standards Institute (ANSI) Z601 standard (IAPMO/ANSI 2016), whereas method 2 used cleaning vinegar (6% acetic acid) to dissolve scale.

Comparing nitric acid (0.1 N) method with cleaning vinegar (6% acetic acid) method

A bench-scale experiment was conducted to illustrate the strength and weakness of the two scale recovery methods. About 210 mg of calcium carbonate scale was placed into 50 mL of cleaning vinegar and 50 mL of 0.1 N nitric acid (Supplementary Fig. S2). After 2 h the samples were filtered through a $0.4 \mu\text{m}$ vacuum filter, and any remaining scale was placed in fresh cleaning vinegar and 0.1 N nitric acid. This process was repeated three times. Samples were collected and analyzed using ICP-MS to determine the amount of scale dissolved in each round.

Importance of scaling location and the effect of phosphate corrosion inhibitors. A practical prototype using phosphate inhibitor was developed that illustrated how profoundly scaling can be affected by distance from the water heater, and to demonstrate that this is a critical performance parameter that might sometimes need to be quantified (Supplementary Data page 5). Results from this experiment showed that in the absence of phosphate, the amount of deposit decreased with distance, whereas with phosphate a peak deposit mass was observed at the midpoint of the pipe (Supplementary Fig. S3), proving that the presence of a constituent such as phosphate could sometimes affect the location and mass of scale deposited in pipes.

Because each scale reduction technology is hypothesized to have different impacts on scaling with distance from the apparatus, it was considered important to at least have a capability to quantify this dimension of performance in the standardized test method. This was done by cutting coil

TABLE 3. EIGHT WATER CONDITIONS AT SATURATION FOR CALCIUM CARBONATE, WHICH WERE TESTED TO DETERMINE AN OPTIMAL STANDARD INFLUENT WATER FOR THE STANDARDIZED SCALING TEST PROTOCOL

Temperature	pH	Hardness [mg/L as CaCO_3]
15	7	349
	7.5	183
	8	100
20	7	320
	7.5	168
25	7	294
	7.5	155

tubing used in each experiment into five 3 m segments to determine the amount of both loose and adherent surface scale formed on each segment of the stainless-steel surfaces.

Water chemistry

Standard synthetic water. The synthetic water developed for the baseline SSTP testing simulated a ground water at saturation with calcium carbonate that developed a very high scaling potential only after it was heated. The goal was to replicate a realistic scaling scenario that had no reason to form a precipitate in the apparatus before the water was heated, thereby eliminating this facet of variability associated with past attempts at standardized testing using supersaturated solutions. To determine the best water chemistry for the SSTP, eight water conditions were initially screened (Table 3). MINIQL⁺ was used to determine the calcium concentrations necessary for each water condition to be at saturation with calcium carbonate.

The synthetic water was prepared by adding sodium bicarbonate, calcium chloride dihydrate, and sodium metasilicate nonahydrate to reverse osmosis-treated water. A water chiller was used as needed, to maintain target temperatures below room temperature (20°C) and submersible pumps were used to mix the water. The pH of the water was adjusted by bubbling in carbon dioxide (to lower the pH) and air (to increase the pH).

Standard synthetic water with orthophosphate. To evaluate the effects of orthophosphate (PO_4) on scaling, the same synthetic water composition with the addition of sodium orthophosphate ($0.2 \text{ mg/L as } \text{PO}_4$) was used. A low dose of orthophosphate was used to show that even a small amount of orthophosphate can have a significant impact on scaling.

Natural water. The SSTP was also tested using naturally hard water from a private well in Blacksburg, Virginia. This water was naturally supersaturated, and it also had a higher total hardness and alkalinity compared with the standard solution used in the laboratory experiments (Table 4).

Scale reduction devices

A marketing study was conducted by the WQRF to identify a list of representative main-stream residential products. One device from each technology category was selected to test in this study (Table 5). We note that the products tested in this study are merely examples of the products in their

TABLE 4. WATER CONDITIONS FOR STANDARD SOLUTIONS AND NATURAL WATER FROM A PRIVATE WELL IN BLACKSBURG, VA

Characteristic or substance	Concentration	
	Standard solutions	Natural water
Hardness [mg/L as CaCO ₃]		
Total	320	410–451
Calcium	320 (100% of total hardness)	215–250 (45% of total hardness)
Magnesium	N/A	170–205 (55% of total hardness)
pH	7	6.7–7.2
Temperature [°C]	20	20–30
Silicate [mg as SiO ₂]	20	11–13
Chloride [mg/L as Cl]	230	40
Sulfate [mg/L SO ₄]	N/A	18
Alkalinity [mg/L as CaCO ₃]	190	355–440
Orthophosphate [mg/L as PO ₄]	N/A	N/A
Saturation ratio ⁴ (Q/K)	1	1.66–2.27

respective technology categories and may differ in material respects from other products in the same technology categories. Products were tested under the conditions matching the manufacturers' use instructions. Performance of different technologies may vary, and the results are provided only for illustrative use of the standardized method. Researchers must follow manufacturers' recommendations for product installation and application to fairly test desired performance.

To demonstrate the reproducibility and capabilities of the standard waters, triplicate tests ($N=3$) were performed for each condition (with and without orthophosphate). The cation exchange softener is a more established technology that indisputably eliminates scaling, and therefore only one replicate ($N=1$) was performed for each of the standard water conditions to confirm the performance of the SSTP in commercial scale reduction treatment devices. To benchmark the SSTP with three emerging commercial scale reduction devices, namely electrochemical deionization, physical magnet, and electric field generator, duplicate tests ($N=2$) were performed for both water conditions (with and without orthophosphate). Duplicate tests of three additional devices (media-induced precipitation, sacrificial media with phosphate, and sacrificial media with citric acid) were performed using the standard solution without orthophosphate.

Another experiment was conducted with all of the scale reduction devices, except the cation exchange water softener, once ($N=1$) using naturally hard water from a well in Blacksburg, VA. The goal was to illustrate that the SSTP could generate reasonable data using a natural water supply,

analogous to the National Sanitation Foundation (NSF)/ANSI 44, IAPMO Guide Criteria-91 (IGC-91), IAPMO Guide Criteria-335, and Deutsche Vereinigung des Gas- und Wasserföhrungsingenieurwesens (DVGW) W 512 standard approaches.

Evaluation of scale reduction devices

The amount of scale recovered after each experiment with a scale reduction device was compared with the amount of scale recovered from the control experiments without treatment to determine the effectiveness of each device. The mean scale recovered from each device was compared with the control experiments. Reduction of scaling was considered significant if the probability value (p -value) was ≤ 0.05 .

Results and Discussion

Selection of a scale recovery method

Two methods of scale recovery were tested, including nitric acid and vinegar.

Using nitric acid to dissolve scale (adapted from IAPMO Z601 standard). Application of the IAPMO Standard method to remove calcium carbonate from heating element and water heater using nitric acid solution was deemed problematic. The heating element rusted and scale remained on the heating element after 24 h in 0.01 N nitric acid solution (Fig. 3a, b). The snake camera demonstrated that scale also remained on the bottom of the water heater (Fig. 3c, d) after 0.1 N nitric acid was used to dissolve the scale.

TABLE 5. TEST CONDITIONS FOR THE STANDARDIZED SCALING TEST PROTOCOL

Device	Standard water (w/o PO ₄)	Standard water (w/PO ₄)	Natural water
No treatment (control)	$N=3$	$N=3$	$N=1$
Cation exchange softener	$N=1$	$N=1$	$N=0$
Electrochemical deionization	$N=2$	$N=2$	$N=1$
Physical magnets	$N=2$	$N=2$	$N=1$
Electric field generators	$N=2$	$N=2$	$N=1$
Media-induced precipitation	$N=2$	Devices not tested due to manufacture constraints	$N=1$
Sacrificial media (phosphate)	$N=2$		$N=1$
Sacrificial media (citric acid)	$N=2$		$N=1$

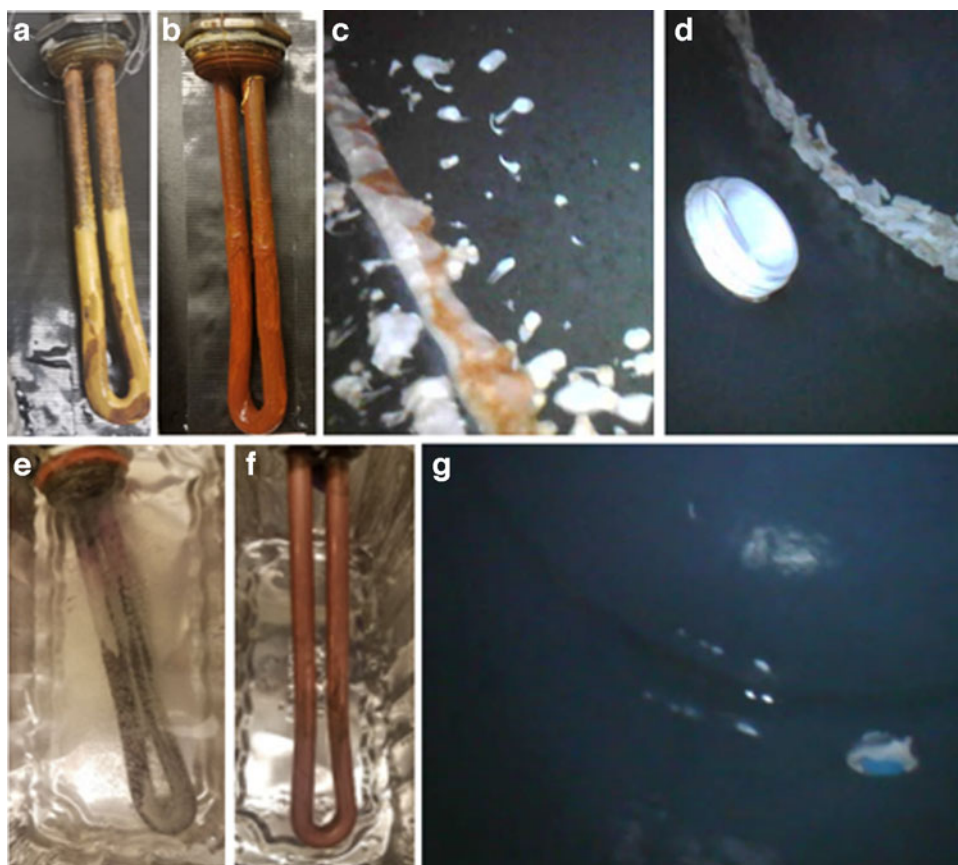


FIG. 3. Photographs of (a) heating element after being placed in 0.01 N nitric acid solution (pH 2) for 2 h, (b) heating element after being placed in 0.01 N nitric acid solution (pH 2) for 24 h, (c, d) inside of heater after 0.1 N nitric acid solution was used to dissolve scale, (e) cleaning vinegar (6% nitric acid) dissolving scale on heating element, (f) heating element after being placed in cleaning vinegar for 1 h, and (g) inside of heater after cleaning vinegar was used to dissolve scale.

Using cleaning vinegar (6% acetic acid) to dissolve scale. The process of removing calcium carbonate from the heating element using cleaning vinegar was more successful than the nitric acid solution. The calcium carbonate was removed from the heating element within 1 h, and there was no visible rust on the heating element (Fig. 3e, f). The snake camera demonstrated that there was no scale remaining at the bottom of the water heater (Fig. 3g), indicating that cleaning vinegar was also successful at recovering the scale from within the water heater.

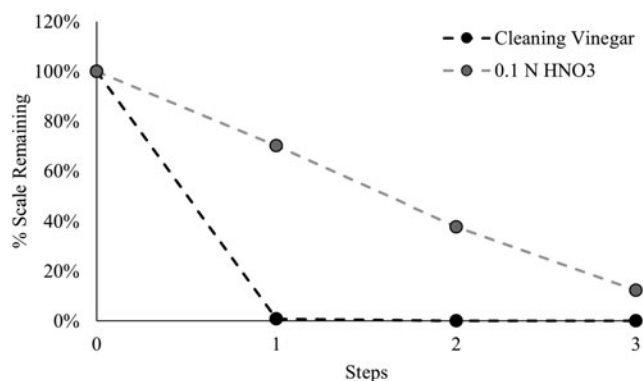


FIG. 4. Scale remaining after being dissolved in cleaning vinegar and 0.1 N nitric acid. The scale was in 50 mL of solution for 2 h each round.

Comparing IAPMO method (0.1 N nitric acid) with SSTP method (cleaning vinegar—6% acetic acid). A bench-scale test confirmed that cleaning vinegar was much more successful at dissolving calcium carbonate scale than 0.1 N nitric acid. After ~2 h in cleaning vinegar <1% of the scale remained, whereas ~70% of the scale remained in the 0.1 N nitric acid solution (Fig. 4). After three cleaning steps in sequence, all the scale was dissolved in the cleaning vinegar solution, but there was still 24 mg of scale remaining in the nitric acid solution. The remaining scale from the nitric acid solution was completely dissolved in 50 mL of cleaning vinegar after ~1 h.

It is unclear why the cleaning vinegar was so much better than nitric acid at recovery of scale. At pH 4–5, Mineql⁺ predicts that 61–84% of the calcium would be complexed by the acetate present in the cleaning vinegar, whereas calcium is not complexed by nitrate. This complexation of calcium might partly explain the enhanced dissolution efficacy of the higher pH vinegar when compared with the lower pH of strong nitric acid.

In addition to being more effective at dissolving scale, cleaning vinegar is cheaper, much less hazardous to personnel, safer to handle, and easier to dispose of.

Water chemistry

Initial experiments to select the standardized water chemistry used a simplified setup using only 20 gallons of water per day without downstream plumbing. As expected,

TABLE 6. WATER CONDITIONS AND SCALE RECOVERED AFTER 5-DAY EXPERIMENT

<i>Water condition</i>			<i>Recovered scale after 5-day experiment [g as CaCO₃]</i>
<i>Temperature</i>	<i>pH</i>	<i>Hardness [mg/L as CaCO₃]</i>	
15°C	7	349	15.6
	7.5	183	6.2
	8	100	2.8
20°C	7	320	15.0
	7.5	168	5.7
25°C	7	294	12.6
	7.5	155	5.7

Bold values indicate water chemistry chosen for the SSTP.

the amount of scale that formed increased with colder temperature and lower pH of the initial water at saturation with CaCO₃ (Table 6). Reducing the influent pH from 8 to 7 had a greater effect on the scaling potential after heating than did reducing the initial temperature of the water from 25°C to 15°C (Table 6).

The water condition selected for the standardized scaling water was pH 7 at 20°C (room temperature) with 320 mg/L hardness. At pH 7, there is not a large difference in scaling at 15°C or 20°C, and it is much easier and more cost effective to run an experiment at room temperature because it does not require refrigeration or water recirculation pumps. Hence, room temperature was adopted as the standard due to considerations of cost and convenience.

Scale recovered from within water heater

Testing demonstrated that the SSTP could produce significant and reproducible scaling in the water heater within the desired short-term experimental time frame of 5 days. The average amount of scale recovered for the triplicate control tests (with no scale reduction device) for synthetic water without orthophosphate and with phosphate was 25.1 and 12.3 g of calcium carbonate, respectively (Table 7 and Supplementary Table S4). The triplicate tests for the control

experiments showed reasonable reproducibility, with a standard error of ± 1.5 mg/L as CaCO₃ for the standard solution and ± 0.3 mg/L as CaCO₃ for the standard solution with orthophosphate.

The vast majority of calcium carbonate precipitation occurred on the heating element, with much of it detaching and falling to the bottom of the heater in all tests under all the standardized test conditions (Fig. 5). Results from recovery of scale within the hot water pipe tubing after the heater (Supplementary Tables S60 and S61) indicated that precipitation on the surface of tubing was not significant (always <0.1% of the total scale formed) when compared with the amount of scale recovered from within the heater.

It is important to note that the SSTP has a significant advantage, due to much shorter 5-day duration than previous standards such as the IAPMO Z601 test, which had a 10-day duration. This translates into only requiring 757 L (200 gal) in the current test versus 18,320 L (4,800 gal) in the IAPMO approach. The amount of scale produced per gallon of water used was calculated and compared with the corresponding values from IAPMO Z601 validation testing, and both approaches were comparable at 0.13 g of CaCO₃ per gallon of water used. But the current approach had an advantage of producing all of the precipitation in the water heater, and was not subject to a variable amount of precipitation before heating as described previously.

Evaluation of scale reduction devices

Illustrative testing revealed that scale reduction for the devices and technologies tested ranged from 0% to 100% under the standardized protocol (Table 7).

Standard synthetic water without PO₄. Results confirmed that the cation exchange water softener and electrochemical deionization device reduced the amount of scale precipitated within the system by >99% (Table 7). The phosphate sacrificial media device and the citric acid sacrificial media device tested were able to reduce scaling by ~50%, while the electric field and physical magnet devices tested in this work reduced scaling by only ~5% (Table 7).

TABLE 7. RECOVERED SCALE FROM WITHIN HEATER

<i>Condition^a</i>	<i>Standard water</i>		<i>Standard water with PO₄</i>		<i>Natural water</i>	
	<i>Mean recovered scale for device [g]</i>	<i>% Reduction compared with control</i>	<i>Mean recovered scale for device [g]</i>	<i>% Reduction compared with control</i>	<i>Recovered scale for device [g]</i>	<i>% Reduction compared with control</i>
Control	25.1		12.3		38.0	
Cation exchange	0.039	99.8	0.02	99.8	<i>Device not tested in Phase 3.4</i>	
Electrochemical deionization	0.24	99.1	0.37	97.0	0.07	99.9
Physical magnet	23.8	5.2	14.4	-16.7	27.5	27.8
Electric field generator	24.0	4.4	11.9	3.5	32.0	15.6
Media-induced precipitation	11.9	52.4	<i>Devices not tested for this water condition due to manufacture constraints</i>		17.1	55.1
Sacrificial media (phosphate)	22.2	11.5			36.0	5.4
Control at pH 7.3	11.5	54.1			7.8	79.5
Sacrificial media (citric acid)	16.1					
	8.5	47.3			10.1	73.4

^apH was 7.0 for all standard water conditions except for the control at pH 7.3 and sacrificial media (citric acid) due to manufacture constraints for this device.

Bold value indicates scale increase rather than scale reduction compared to the control condition.

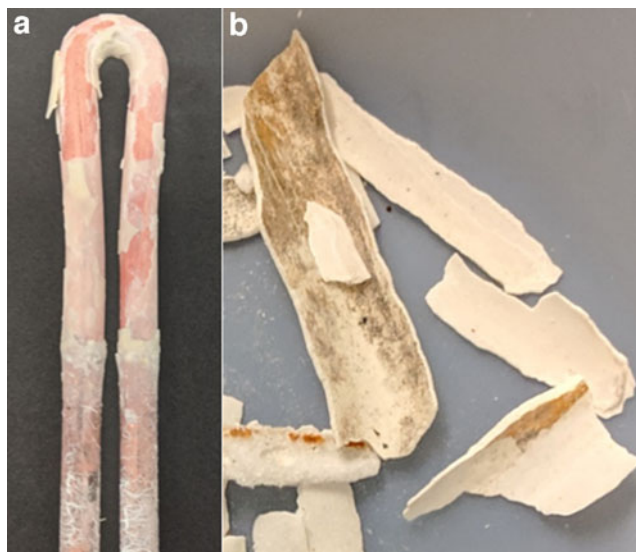


FIG. 5. Scale that (a) formed on the heating element and (b) fell from the heating element and accumulated on the bottom of the tank.

The testing approach could demonstrate that the effectiveness of the specific media-induced precipitation device tested in this study decreased over the 5-day test duration. In the first experiment there was ~50% reduction in scale, whereas the second experiment reusing the device, there was only a 11% reduction in scale (Table 7). Daily samples (Fig. 6) showed that the device was initially reducing the amount of calcium in the water (to <200 mg/L as CaCO₃ on Day 0 of Experiment 1). However, by Day 4 of Experiment 1, the device stopped removing calcium from the water, and this continued to be the case in Experiment 2. The findings from the media-induced precipitation experiment illustrate the importance of collecting daily samples (Fig. 6) in some cases, to better understand how performance of each device may be changing over time. In other words, some devices had completely stopped working after as few as 4 days in the standardized water, demonstrating the usefulness of third-party performance verification and testing.

When mean scale recovered from each device was compared with the control experiments for the standard solution experiments, the electrochemical deionization de-

vice and the phosphate sacrificial media device significantly reduced scaling in the standard water experiments (Student's *t*-test *p*-value <0.05; Supplementary Table S3). Since only one experiment each was performed for the illustrative experiment with cation exchange and the control experiment for the pH 7.3 condition for the citric acid sacrificial media device, statistical analysis could not be conducted for either condition.

Standard synthetic water with PO₄. The amount of scaling was significantly reduced (*p*-value = 0.0067) when a very low dose of inhibitor (0.2 mg/L PO₄) was added to the standard solution (Supplementary Table S4). The amount of scale recovered from the triplicate testing of the control condition with orthophosphate was reasonably reproducible, providing a 95% confidence interval of 9.6–14.9 g calcium carbonate.

Of the devices tested using the standard water with orthophosphate, we found that the electrochemical deionizer was the only device to significantly reduce scaling (Supplementary Table S5).

The cation exchange, electrochemical deionization, and electric field generator all performed similarly in the standard solution with orthophosphate added as corrosion control, compared with how they performed in the standard solution without orthophosphate (Fig. 7). Hence, the presence of orthophosphate corrosion inhibitor did not have an impact on the performance of these devices. The physical magnet device caused slightly more scale to precipitate within the heater for the water that had orthophosphate than the water without orthophosphate, although this slight increase was not statistically significant (Fig. 7 and Supplementary Table S5).

Natural water. Illustrative results obtained using the natural water compared favorably with those with the synthetic water, although some devices were more effective when tested with the natural water (Fig. 7). This might be because the natural water was supersaturated with calcium carbonate ($\Omega = 1.66\text{--}2.37$) and that might be a prerequisite for satisfactory performance of some technologies.

The electrochemical deionization and sacrificial media (both phosphate and citric acid) devices were able to reduce the amount of scale precipitated within the system by >70% when compared with the control condition without any treatment. A slightly greater reduction in scaling was

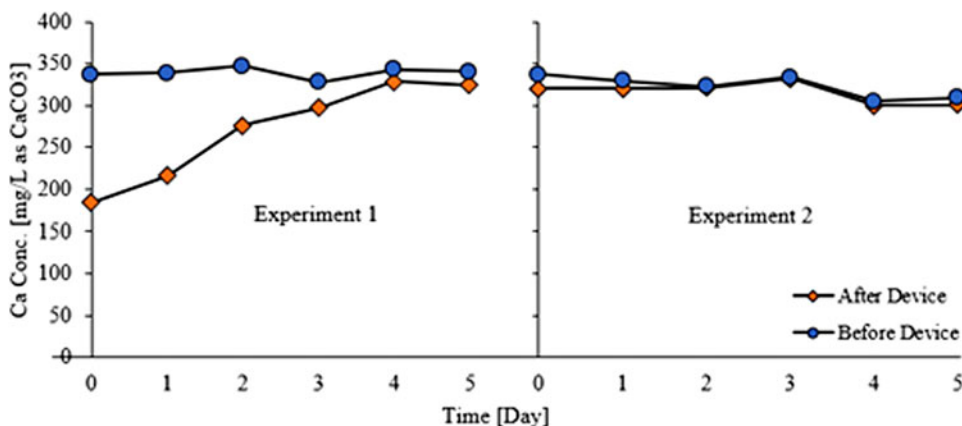
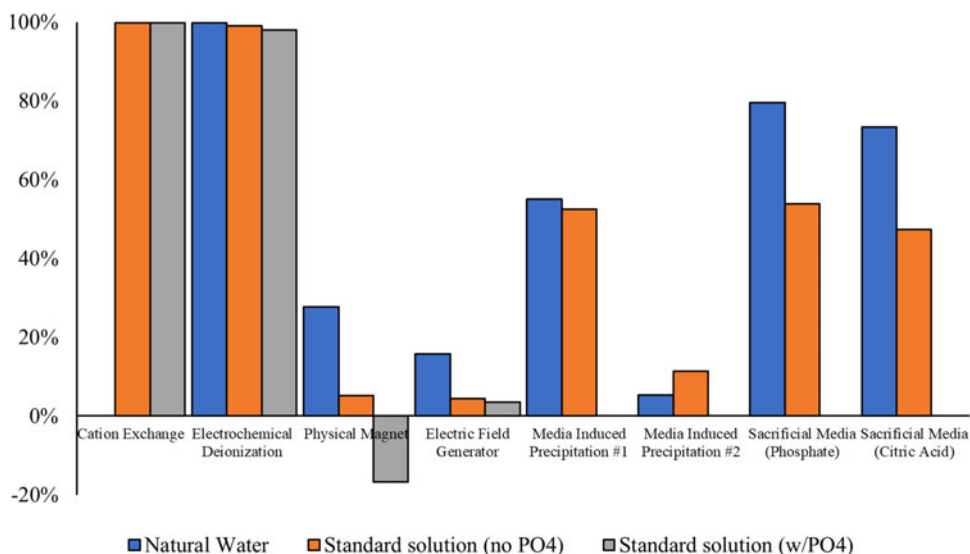


FIG. 6. Calcium concentration in water before and after media-induced precipitation device over time.

FIG. 7. Percentage scale reduction for each device in natural water (blue), standard solution without PO₄ (orange), and standard solution with PO₄ (gray).



achieved from the physical magnet and electric field generator for the natural water testing than the synthetic water testing. That is, the physical magnet reduced scaling by 27.8% in the natural water and 5.2% in the standard solution, whereas the electric field generator reduced scaling by 15.6% in the natural water and 4.4% in the standard solution conditions. This suggested for these technologies that they were 3.5–5 times more effective in the natural water than in the standard solution (Table 7).

The results for the media-induced precipitation device in the natural water were similar to the results for the media-induced precipitation device in the standard solution. The device reduced the amount of scaling in the first 5-day experiment by >50% in both cases, but only reduced scaling by 5.4–11.5% for the second 5-day experiment (Table 7).

Future research and modifications

The effectiveness of devices will vary depending on the specifics of water chemistry. Variations in the baseline SSTP protocol and water recipe presented herein can be made in future work, to systematically sort out how effective each device is and under what circumstances. For instance, future modifications could be made to the SSTP protocol to test the impact of minor and major trace elements in the water versus the control, suspended particulates, the effect of flow rate, or waters supersaturated with CaCO₃ in the cold water intake. All of these factors are suspected to affect the rate of scaling, location of deposits, and the relative efficacy of the different technologies.

Conclusion

The SSTP developed in this study was able to reproducibly and quickly evaluate the performance of scale reduction technologies in one reproducible water chemistry. The reproducibility of the standardized water and test protocol means that these tests can be done in any laboratory, which would allow testing and verification of performance claims, and lead to improved understanding of antiscaling mechanisms. The SSTP protocol can be modified and applied to natural water and synthesized water with corrosion inhibitors.

Authors' Contributions

C.D. executed the project, conducted the experiments, and led the write-up for this article. F.W. cowrote the proposal for funding this experiment and contributed to the introduction section of this article. M.E. conceived the experiments, advised on the work, and coauthored the work.

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Author Disclosure Statement

No competing financial interests exist.

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Supplementary Material

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