

Molybdate and Non-Molybdate Options for Closed Systems – Part I

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This article was written with the input of the AWT Technical Committee. The article is designed to provide information regarding the use of molybdate and non-molybdate chemical treatments for closed cooling water systems.

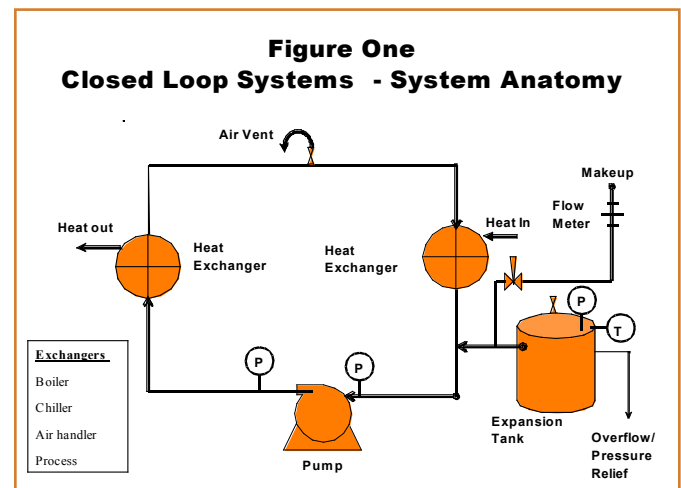
Abstract

Treatment of closed loops, since the enactment of regulations restricting the use of chromates, has typically relied on the use of a select few list of corrosion inhibitors. Sodium molybdate has become a particularly common replacement for chromate because: 1.) It is a very good low carbon steel corrosion inhibitor; 2.) It is relatively persistent unless water losses occur; 3.) There is a reliable field test readily available; and 4.) Unlike sodium nitrite, sodium molybdate does not contribute nutrients, which can exacerbate microbiological activity in a closed loop. However, over the previous five years, there has been a slow but consistent trend by municipal wastewater treatment facilities to restrict, and in some cases ban, the discharge of sodium molybdate to the sanitary sewer. Moreover, recently the water treatment industry has had to deal with a significant rise in the purchase cost of sodium molybdate raw material. These market trends have forced water treaters to evaluate alternative treatment methods. The goal of this article is to review the treatment of closed loops with an emphasis on corrosion inhibitor selection and use. Consequently, molybdate alternatives can be evaluated.

Section One Closed System Design & Operation

Closed recirculating systems or closed hydronic loops utilize a water-based solution to transfer heat. The most common distinction defining a closed system is the fact that the method of cooling is non-evaporative. Minimal

water loss/makeup requirement and minimal air contact are two additional conditions typically associated with closed loops. Closed systems are so diverse in application that it can be difficult to suggest a standard set of operating conditions and design. Figure 1 shows a simple illustration conceptualizing the closed loop system.



The heat transfer will typically be indirect utilizing some type of heat exchanger. One or more exchangers will take on heat and one or more heat exchangers will give off heat, thus maintaining heat balance. Heat transfer surfaces must be maintained in a clean condition to ensure efficient and reliable operation. The distribution system can be simple as shown above or it can be com-

plex; for example, a high rise HVAC distribution system can be complex since it is integrated with the building infrastructure making maintenance/pipe replacement complicated and expensive. Materials of construction can be diverse, but typically the materials are steel piping and tanks, and stainless steel or copper/copper alloy exchangers. Aluminum, galvanized steel, and other exotic alloys, can be used as well. In many cases, these systems are designed with a surge tank or expansion tank. This tank can operate at atmospheric pressure or at elevated pressure and utilize a pressure relief device to prevent over-pressurization. Air vents are used to help remove oxygen and other non-condensable gases from the system at startup and whenever water is added to the system. Pumps are used to recirculate the water-based solution throughout the distribution system.

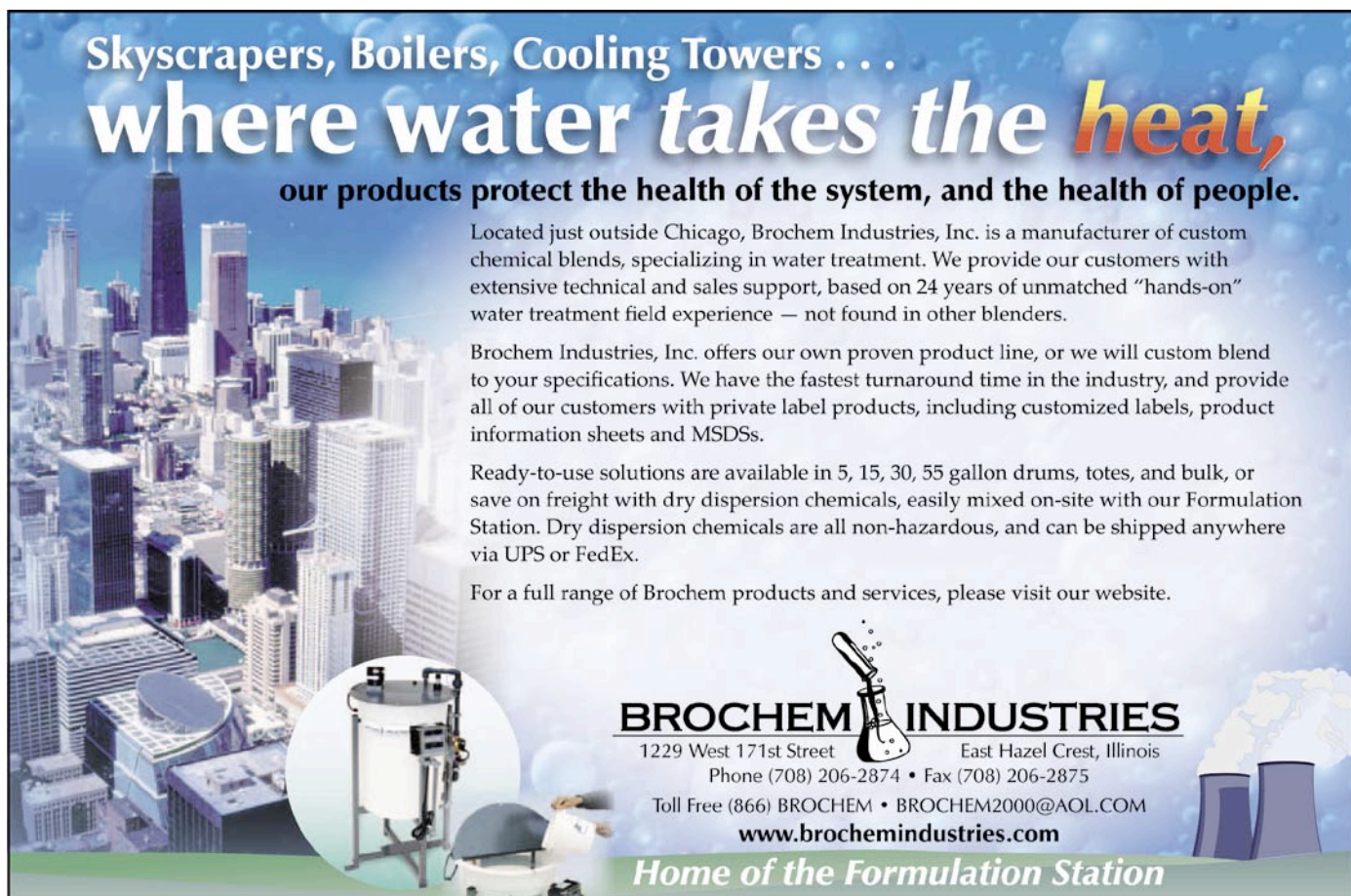
Operating conditions associated with closed loops, such as, little to no evaporation (i.e., no cycling of chemistry), minimal water losses and minimal air contact, should allow for near zero corrosion and fouling rates to be achieved. However, these operating conditions are not always present and consequently, this can significantly influence the

treatment program selection process. Below are some key operating and design elements that can greatly influence the choice of chemical treatment:

- Degree of Air Contact or Ingress (atmospheric vs. pressurized)
- Water Loss (<1 %/month vs. >1 %/month)
- Temperature (chilled water vs. hot water)
- Heat Transfer Solution Chemistry (conductivity sensitive loops, brine loops, glycol loops, etc.)
- Makeup Water Chemistry (hardness, suspended solids, microbiological activity, etc.)
- Materials of Construction (steel, copper, aluminum, other)
- System Cleanliness (new, clean, fouled)
- System Size (volume, large, small, etc.)

Degree of Air Contact or Ingress & Water Loss

It is expected that a pressurized closed loop with minimal water loss will have minimal oxygen content after operating for a period of time. Generally, the tighter the



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system, the easier it is to maintain effective treatment. It is important to understand the quantity of makeup water and the amount of oxygen ingress that may be occurring (versus what is expected), as these parameters will potentially affect treatment cost and performance. Obviously, if water loss and oxygen ingress can be reduced, this should be done. In order to effectively track water losses, each closed loop should have a makeup flow totalizer and the meter reading should be monitored routinely. Water losses can occur for various reasons, such as, pump seal leakage(s), improper level control or improper pressure control, invasive system maintenance or intentional water draw. Air in-leakage can be associated with makeup water ingress or it can occur in the absence of water losses such as with faulty air vents. Air and water ingress can affect the selection of a closed loop treatment inhibitor. For example, silicates can be an excellent choice for high makeup systems because they are low cost (especially in comparison to molybdate treatment), can function in the presence or absence of oxygen, protect multiple metallurgies and do not contribute nutrient that can exacerbate microbiological activity.

Vapor phase corrosion inhibitors (VCI) can be an excellent choice where oxygen ingress is a particular issue, as the VCI products protect the metal surface in the water, water-vapor interface and in the vapor phase. On the flip side, nitrites may be avoided in high makeup systems where microbiological activity is difficult to control or where oxygen ingress is notable.

Temperature and Chemistry

Closed loops can be categorized by the system's operating temperature; for example, chilled water systems will typically operate at or below room temperature (i.e. <72 °F). Hot water loops can be further categorized by temperature. For example, hot water systems operating at or well above 212 °F may be referred to as high temperature hot water systems, while moderate temperature hot water loops will tend to operate below 212 °F. Water temperature, besides impacting the corrosion potential, can dictate the preference and limitation of various treatments. For example, under hot water conditions nitrites are particularly appealing because they are effective in the absence of oxygen, do not contribute to sludge loading, are rela-

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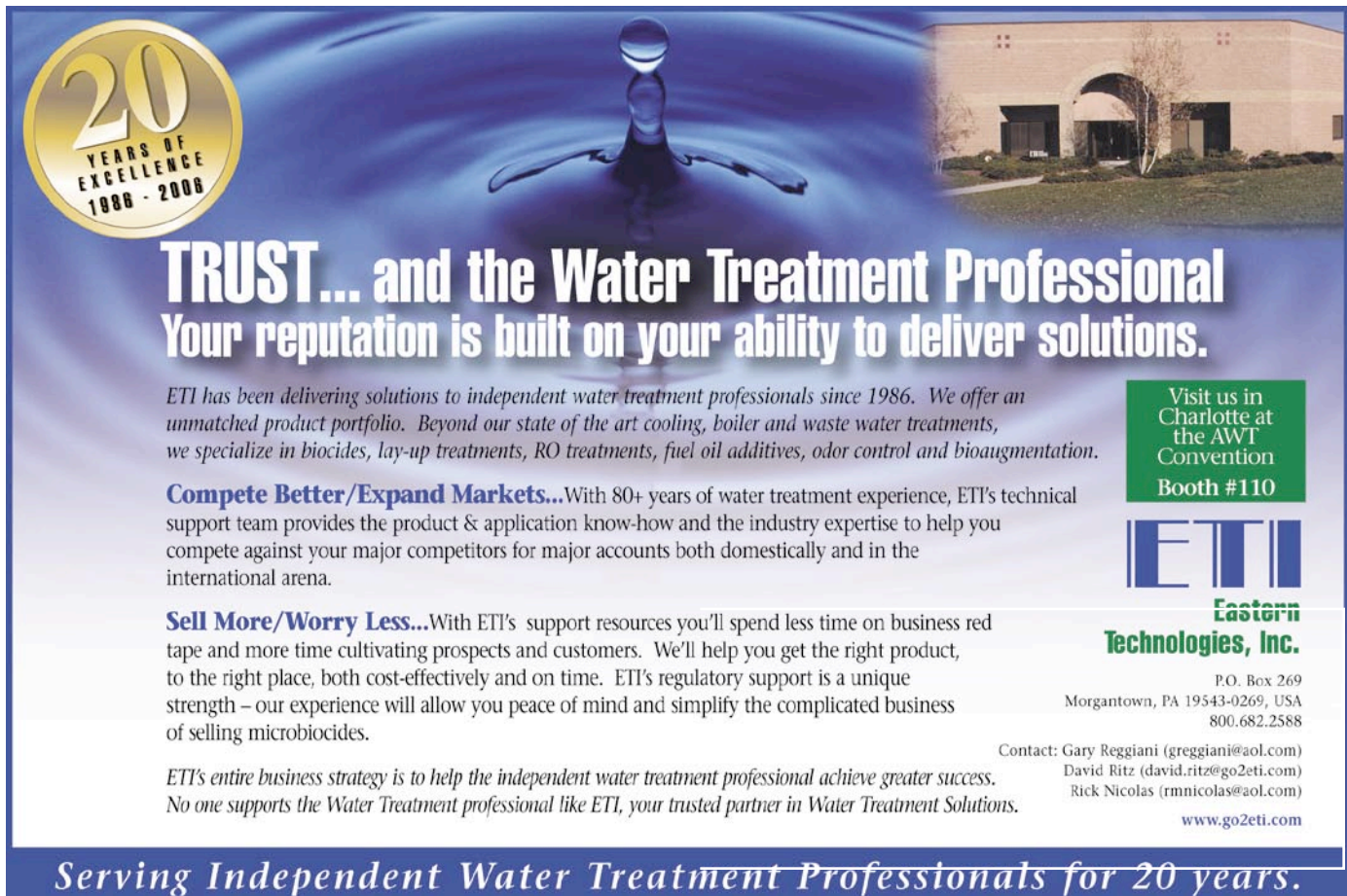
tively inexpensive and due to the water temperature, there is little concern for microbiological control. Sulfites may be used instead of nitrites for high temperature loops. Silicates are often avoided at temperatures greater than 180 °F due to concern with deposition. Under chilled water conditions, nitrites (while still cost effective) lose some of their appeal since they contribute nutrient that can aid microbiological growth. Silicates and particularly molybdate are common alternative choices to nitrite when microbiological issues are chronic. Dual temperature loops will operate at times in the chilled water mode (typically during the cooling months) and at times in the hot temperature mode (typically during the heating months). Depending on the piping configuration (i.e., two pipe versus four pipe system), part or nearly all of this system may utilize the same recirculating water for

The reason that ethylene or propylene glycol is added to a closed system is to freeze-proof the system.

both operating conditions - this will need to be factored when deciding upon a treatment.

The recirculating water chemistry (excluding the treatment chemicals added) should be similar or identical to the makeup water source chemistry since little to no evaporation is expected. Recirculating water chemistry requirements can drive the treatment selection process. For example, conductivity sensitive closed loops will typically require all-organic based treatments that can provide effective performance while contributing little conductivity to the water. For brine solution loops (specifically calcium-based brine), one must consider the potential for calcium deposition as well as corrosion. Consequently, these treatments must perform in aggressive conditions at only slightly alkaline pH (preferably <8.5). Buffered molybdate treatments have been used with some success and newer organic-phosphate treatments have had success.

For glycol-based loops, one must consider if the glycol is preformulated with treatment and with what type of treatment. Treatment (preformulated or otherwise) should



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consider the quality of solution water used with the glycol and the materials of construction. Glycol manufacturers will typically provide water quality specifications for the dilution water for their glycol. Hardness in particular should be scrutinized when using monophosphates (typically dipotassium phosphate/DKP), although polymers have increased the tolerance for hardness.

The reason that ethylene or propylene glycol is added to a closed system is to freeze-proof the system. Examples of systems requiring freeze proofing include systems in which pipes are exposed to cold air and ice and snow melting systems. Glycol concentrations can range from 20 % to 50 %, with the actual glycol concentration of a particular system being selected based on the desired freeze point of that system. For a given freeze point, a lower percentage of ethylene glycol is needed than propylene glycol. However, as indicated in Table 1, ethylene glycol is toxic to humans. Interestingly, the oral LD₅₀ for rats is very high for ethylene glycol indicating low toxicity to rats, yet it is very toxic to humans. (Technical ed. LD or lethal dose is a measure used in pharmacology to express the percentage of a population killed by a dose of the substance being studied. The measurement is often given as a subscript. For example, the potency of a drug or pesticide is commonly expressed by stating the size

However, as indicated in Table 1, ethylene glycol is toxic to humans. Interestingly, the oral LD₅₀ for rats is very high for ethylene glycol indicating low toxicity to rats, yet it is very toxic to humans.

Table 1. Toxicity of Closed System Treatment Components

Test Material	Oral LD ₅₀ (rat mg/Kg)	Dermal LD ₅₀ (rabbit mg/Kg)	Inhalation LC ₅₀ (rat as indicated)
Sodium molybdate dihydrate	4,233	>2,000	>1.93 mg/L/4hr
Sodium nitrite	180	N.A.	5,500 µg/m ³ /4hr
Sodium metasilicate	800	N.A.	N.A.
Sodium nitrate	1,267	N.A.	N.A.
Hydroxyphosphonoacetic acid (HPA), 50 %	2,750	N.A.	N.A.
Tris-(aminopentamethylene-carboxylic acid)-triazine, 63 %	>5,000	N.A.	N.A.
Diethylhydroxylamine (DEHA)	2,190	1,300	3,140 mg/L/4hr
VCI/Organic filmer blends	See manufacturer for data.		
Dipotassium phosphate (DKP)	>500	>300	N.A.
Sodium tolyltriazole (TTNa), 50 % Tolyltriazole (TTA), 100 %	920, male 640, female (TTNa, 50 %)	>2,000 (TTA, 100%)	N.A.
Benzotriazole (BT)	560	>2,000	1,910 mg/m ³ /3hr
Sodium tetraborate pentahydrate	3,200-3,400	>2,000	N.A.
60/40 AA/AMPS* Copolymer, 28 %	>5,000	>2,000	N.A.
AA/AMPS/SS**	N.A.	N.A.	N.A.
Ethylene glycol	6,000-13,000	>22,270	>3.95 mg/L/7hr aerosol
Propylene glycol	20,000-34,000	>10,000	N.A.

* 60/40 AA/AMPS: 60 wt % Acrylic Acid / 40 wt % acrylamidomethylpropylsulfonic acid

** AA/AMPS/SS: Acrylic acid/acrylamidomethylpropylsulfonic acid/sulfonated styrene

of the LD₅₀ dose: the amount of the substance that kills 50% of the test population.) This contradiction illustrates that sometimes the results of animal toxicity testing cannot be extrapolated to humans. In contrast, propylene glycol is much less toxic to humans and therefore the use of propylene glycol is increasing.

Glycol concentrations should not be allowed to fall below 20%. One reason is that glycols become nutrients for microbes when the concentration falls below 20%, whereas glycols become biostatic at concentrations above 20%. A second reason is that freeze protection will not be adequate at low levels of ethylene or propylene glycols. Graphs or charts of glycol concentration versus freeze point are available from glycol manufacturers and can be used to select the correct concentration to obtain a desired level of freeze proofing. Inhibited glycols contain dipotassium phosphate for buffering the system at pH 9.0 to 10.5 and for preventing low carbon steel corrosion. Molybdate, nitrite, or molybdate/nitrite programs are often added to uninhibited glycol to control low carbon steel corrosion. Azoles are usually added for controlling yellow metal corrosion and for complexing any dissolved copper impurities.

Materials of Construction

It is critical to understand the materials of construction present in the closed loop when considering the chemical treatment program. When chromates are used, there was little need for multi-component inhibitor formulations regardless of the material of construction. With the elimination of chromates there was a need to consider formulating multiple inhibitor components based on material of construction. Today it is very common to use multicomponent formulations to optimize the protection of multimetal systems. Certain metallurgies, such as aluminum will require special consideration in choosing a treatment program. The use of azole is common and is recommended for almost all closed loop treatment formulations just in case there is copper metallurgy or in the event that copper is a contaminant to the system. Table 2 summarizes a list of commonly used inhibitors and the applicable metallurgies they protect.

System Cleanliness

To achieve effective treatment performance, the system metal surfaces must be clean. Use of an effective clean-



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ing solution along with side stream filtration should be considered for new and old systems alike that may require cleaning. Special care should be given to addressing microbiological control prior to treatment with nitrite-based formulations and then again as needed.

Treatment formulations may include deposit control inhibitors to help maintain clean surfaces and to prevent underdeposit attack. The installation of side stream filtration is a relatively low cost add-on that can pay dividends

down the road. If filtration equipment design incorporates backwashing, be sure that fresh makeup water is used and not closed recirculating water.

System Volume

Knowledge of system volume is critical to ensuring adequate chemical dosing. Additionally, the size of the system may impact on the choice of treatment, particularly if the system volume is significant and/or has notable water loss. Water losses, barring uncontrolled and controlled

Table 2. Recommended Inhibitor Levels for Closed Systems

Inhibitor	Metal(s) Protected	Typical pH Range****	Active Dose (mg/L)
Molybdate, Alone	Fe, Al	8.5-10.5 Fe 7.8-8.3 (<9.0) Al	150-1,000 as Na ₂ MoO ₄ *, ref. 3 (177-777 as MoO ₄ ⁻²) (70-466 as Mo ⁺⁶) ⁴
Nitrite	Fe	8.5-10.5 Fe	500-1,000 NO ₂ ⁻ or 750-1,500 as NaNO ₂ , ref. 1 600-1,200 NO ₂ ⁻ or 900-1,800 as NaNO ₂ , ref. 2 467- 800 NO ₂ ⁻ or 700-1,200 as NaNO ₂ , ref. 4
Molybdate/Nitrite	Fe, Al	8.5-10.5 Fe 7.8-8.3 (<9.0) for Al	50:50 - 60:40 Na ₂ MoO ₄ : NaNO ₂ Ratio, ref. 6 150+150 – 400 + 400 Na ₂ MoO ₄ + NaNO ₂ is a typical range Low Molybdate: 161 Na ₂ MoO ₄ + 525-750 NaNO ₂ , ref.1
Nitrite/Nitrate	Fe + Al	7.8-8.3 (<9.0) for Al	500-1,000 NO ₂ ⁻ + 500-1,000 NO ₃ ⁻ , ref. 2 (750-1,500 as NaNO ₂ + 685-1,371 NaNO ₃)
Silicate	Fe**, Al, Cu	8.5-10.5 Fe 7.8-8.3 (<9.0) for Al	50-100 as SiO ₂ alone for multimetals, ref. 1 10-25 as SiO ₂ adjunct for Al and Cu alloys
Hydroxyphosphonoacetic acid (HPA)	Fe	8.5-10.5 Fe	50-200 HPA, ref. 4 (30-122 Organic Phosphate as PO ₄ ⁻³)
Hydroxyphosphonoacetic acid (HPA)/Tris-(aminopentamethylene-carboxylic acid)-triazine***	Fe	8.5-10.5 Fe	100-150 actives, ref. 5 (35-50 Organic Phosphate as PO ₄ ⁻³)
DEHA	Fe	9.5-10.0 Fe	200 mg/L initially; 125 mg/L maintenance is a typical range
VCI/Organic Filmer Blends	Fe, Cu, Al	8.5-10.5 powder 8.0-9.0 liquid	1000+ mg/L for powder products 2000+ mg/L for liquid products
Orthophosphate (Glycol Systems)	Fe	9.0-10.5 Fe	1,000- 5,000 as PO ₄ ⁻³ , ref. 4
TT/BT	Cu	8.5-10.5 Cu with Fe	10-50, ref. 1
Dispersants	All surfaces	7.5-10.5 Fe 7.8-8.3 (<9.0) for Al	10-20 mg/L (typical)

* Actual dose dependent on electrolyte concentration of closed system and requires water to have ≥1 mg/L D.O.

** No heat load with soft or deionized water or high temperature with deionized water only

*** 1:1 Ratio

**** Ref. 4 for Fe and Cu

leaks, should be essentially zero for most systems.

Consequently, in tight systems, the initial cost of a well-designed treatment program will pay dividends in the long run – do not compromise. System volume measurements and trending of water losses are essential pieces of information. This data can be achieved by relatively simple methods that will not be addressed in this article. When assessing system volume, be sure that the entire volume is accounted for, i.e., parts of the system that may be idle during volume assessment should be operated and/or accounted for. ♦

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