Case Study – Fargo, North Dakota: Hydrogen Peroxide for Regeneration of Ferrous Chloride, an Innovative Approach to Hydrogen Sulfide Control

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ABSTRACT

Historically, ferrous chloride ($FeCl_2$) injection has been utilized to successfully control odors and corrosion within the sanitary sewer collection system of Fargo, North Dakota. However, increased odor complaints prompted an evaluation of the odor conditions present in the sanitary sewer system. Study results indicated that the current $FeCl_2$ dosing rate was insufficient to control odors. In response, a number of liquid phase odor control treatment alternatives were evaluated. Due to the existing $FeCl_2$ injection program, iron regeneration and improved odor control were realized through the innovative use of hydrogen peroxide. A program was initiated to demonstrate the efficacy of the proposed treatment strategy on a full-scale level. In response to improved sulfide treatment, reduced $FeCl_2$ injection rates, and economic benefits realized during the demonstration program, the City of Fargo elected to incorporate the technology as part of their upcoming collection system improvements.

KEYWORDS: Odor Control, Hydrogen Peroxide, Ferrous Chloride, Hydrogen Sulfide, Dissolved Sulfide.

INTRODUCTION

The City of Fargo, North Dakota, wastewater treatment system serves the communities of Fargo, Frontier, Prairie Rose, Briarwood, Highland Park, North River, Oxbow, Reile's Acres, and rural Southeast Cass developments. Fargo operates an advanced wastewater treatment facility (WWTF) with a design average daily flow of 56,780 m^3/d (15.0 mgd) and a peak flow capacity of 98,420 m^3/d (26.0 mgd). The liquid treatment processes includes influent pumping, screening, grit removal, pre-aeration, primary clarification, secondary trickling filters, intermediate clarification, tertiary trickling filters, final clarification, chlorine disinfection, and a membrane/reverse osmosis (RO) effluent reuse facility that supplies water to industrial customers. The WWTF continually discharges treated effluent to the Red River of the North.

Due to the lack of topography in the Red River Basin and the size of the collection system area, the City relies on numerous lift stations, forcemains and long gravity pipelines known as interceptors to convey wastewater to the treatment facility. The sanitary sewer collection system contains approximately 580 kilometers (360 miles) of gravity and pressure pipe including 59 sanitary lift stations. Because of the elongated detention times associated with the Fargo collection system anaerobic conditions develop resulting in odor and corrosion.

The most identifiable culprit contributing to odors and corrosion in wastewater collection systems is hydrogen sulfide (H_2S). H_2S is notable for its rotten egg smell, toxicity and corrosive effects. H_2S is formed when dissolved oxygen is depleted in water resulting in anaerobic conditions. In these anaerobic conditions, microbes reduce naturally occurring sulfates to sulfides in the anoxic slime layer that tends to accumulate on pipe walls and in sludge deposits found in pipe inverts. This reduction is expressed by the following reaction (WEF, 2004):

$$SO_4^{-2} + 2C + 2H_2O \xrightarrow{bacteria} 2HCO_3^{-} + H_2S \qquad (1)$$

In wastewater, the soluble, aqueous form of sulfide is referred to as dissolved sulfide (DS). Dissolved sulfides have no odor in the aqueous form; however they can leave the aqueous phase and enter the atmosphere as H_2S gas (USEPA, 1974). H_2S is the only species of sulfide directly capable of causing odors and corrosion. For this reason, DS concentration is often used as a means to measure the potential for odor in wastewater. To control H_2S release, the USEPA recommends maintaining DS concentrations at or below 1.0 mg/L (USEPA, 1985).

Ferrous chloride ($FeCl_2$) is an iron salt that facilitates the removal of DS through chemical suspension and precipitation. When $FeCl_2$ reacts with DS it forms an insoluble precipitate known as iron-sulfide (FeS) (WEF, 2004). This reaction prevents the release of H_2S to the atmosphere thereby controlling odors and reducing the probability for corrosion (USEPA, 1991). The precipitates are held in suspension by the wastewater's velocity as they travel through the sanitary collection system. Once at the WWTF, FeS settles and collects with other sludges at the bottom of clarification units (WEF, 2004).

In the late 1980s, Fargo experienced "crown rot" by way of H_2S corrosion in concrete collector pipelines. As a result, bench scale studies were performed to find a suitable liquid phase approach that would control corrosion within the collection system. $FeCl_2$ was found to be effective at reducing DS and was the most economical alternative for corrosion control. For these reasons, a permanent $FeCl_2$ injection station was constructed in 1992 to treat sulfides in the collection system and prevent infrastructure deterioration. The secondary benefit of $FeCl_2$ injection was odor control along the collector routes.

Although the existing $FeCl_2$ approach continued to provide corrosion control, increased odor complaints from residential areas in 2006 prompted City of Fargo officials to employ the services of an engineering consultant (Ulteig Engineers, Inc., Fargo, North Dakota, USA) to evaluate the odor conditions present at the municipality's WWTF and in the sanitary sewer collection system. The 2006 odor study results illustrated that the current $FeCl_2$ dosing strategy was insufficient to control sulfides to acceptable levels ($\leq$ 1.0 mg/L DS) throughout the collection system and at the WWTF headworks. Furthermore, elevated levels of DS and H_2S were detected in the downstream reaches of the City's largest sanitary sewer interceptor, the West Side Interceptor (WSI).

In response, a number of liquid phase treatment alternatives were evaluated, in terms of effectiveness and monetary considerations, to enhance sulfide control. The technologies considered included increased $FeCl_2$ dosing, other metal salts, nitrate salts, and oxidants.

Because the City of Fargo utilized an iron salt for sulfide control, iron regeneration and improved odor treatment were possible through the innovative use of hydrogen peroxide (H_2O_2).

H_2O_2 is an oxidant capable of reducing DS concentrations in wastewater to non-detect levels (USEPA, 1991). H_2O_2 oxidizes DS to elemental sulfur preventing the formation of additional sulfides downstream. Furthermore, the byproducts of H_2O_2 are harmless to the environment (USEPA, 1985). Used on its own H_2O_2 does not have the capacity for residual sulfide treatment and therefore multiple injection points are required for total system control. However, in conjunction with iron salts, H_2O_2 has the ability to not only oxidize DS at the point of injection, it also releases iron previously bound by the reaction with DS. The regenerated iron is then available to react with additional sulfides downstream thereby providing residual DS control (Neofotistos; *et al.*, 2006). These reactions are illustrated below:

Ferrous Chloride and Hydrogen Sulfide Reaction:

$$2H_2S + 2FeCl_2 \rightarrow 2FeS + 4HCl \qquad (2)$$

Iron Regeneration and Sulfur Formation via Hydrogen Peroxide:

$$2FeS + 3H_2O_2 \rightarrow 2S_o + 2Fe(OH)_3 \qquad (3)$$

Subsequent Iron and Hydrogen Sulfide Reactions:

$$2Fe(OH)_3 + 3H_2S \rightarrow S_o + 2FeS + 6H_2O \qquad (4)$$

Used in the iron regenerative mode, the required H_2O_2 injection is reduced by approximately 75%, when compared to the H_2S preventative dose. In general, the lower dosing rate of H_2O_2 is more economical than increased $FeCl_2$ injection (Walton, J; *et al.*, 2005). In addition to economic considerations, lower $FeCl_2$ dosing reduces solids loading to the WWTF (lower sludge production) and diminishes the potential for alkalinity concerns (impedance of biological treatment) associated with the over-use of metal salts (WEF, 2004). The innovative process by which H_2O_2 is utilized to regenerate iron salts (such as $FeCl_2$) for sulfide control is known as Peroxide Regenerated Iron - Sulfide Control, or PRI-SC (Walton, J; *et al.*, 2003).

Based on the anticipated economic savings and improved treatment efficiency, hydrogen peroxide was recommended as a means to further reduce sulfides, regenerate spent $FeCl_2$ for residual sulfide treatment, and prevent the exacerbation of suspended solids loading to the WWTF headworks.

OBJECTIVES

Following the recommendation, a program was initiated to demonstrate the efficacy of the proposed PRI-SC treatment strategy on a full-scale level. The primary objectives of the demonstration program were focused on the following:

- a proof of concept evaluation of the PRI-SC technology for collection system sulfide control,
- a fundamental comparison to the current $FeCl_2$ treatment program, in terms of performance, operational efficiency and cost benefit, and

- a treatment target of dissolved sulfide concentrations reduced to less than 1.0 mg/L in the collection system.

METHODOLOGY

Approach

From July through October of 2007, the PRI-SC demonstration program was conducted along the West Side Interceptor. During the demonstration process, US Peroxide (US Peroxide, LLC, Atlanta, Georgia, USA) was contracted to supply hydrogen peroxide, temporary chemical storage, peroxide injection equipment, data collection, and summary reporting. Ulteig Engineers directed and supervised US Peroxide, performed data analysis, carried out supplemental field testing during the demonstration period, as well as prepared the final report.

The approach involved the utilization of the City's existing $FeCl_2$ injection station as the source of iron. H_2O_2 was added at a downstream lift station (where residual $FeCl_2$ was diminished) for regeneration purposes. The program was demonstrated using H_2O_2 (50% solution) injection rates varying between 2.2 to 3.5 milliliters per second (mL/s) and two $FeCl_2$ (25% solution) feed rates of 10.5 and 12.6 mL/s. In comparison, the historical $FeCl_2$ treatment program consisted of a flat dose of 17.9 mL/s. Figure 1 below is a flow diagram illustrating the demonstration program.

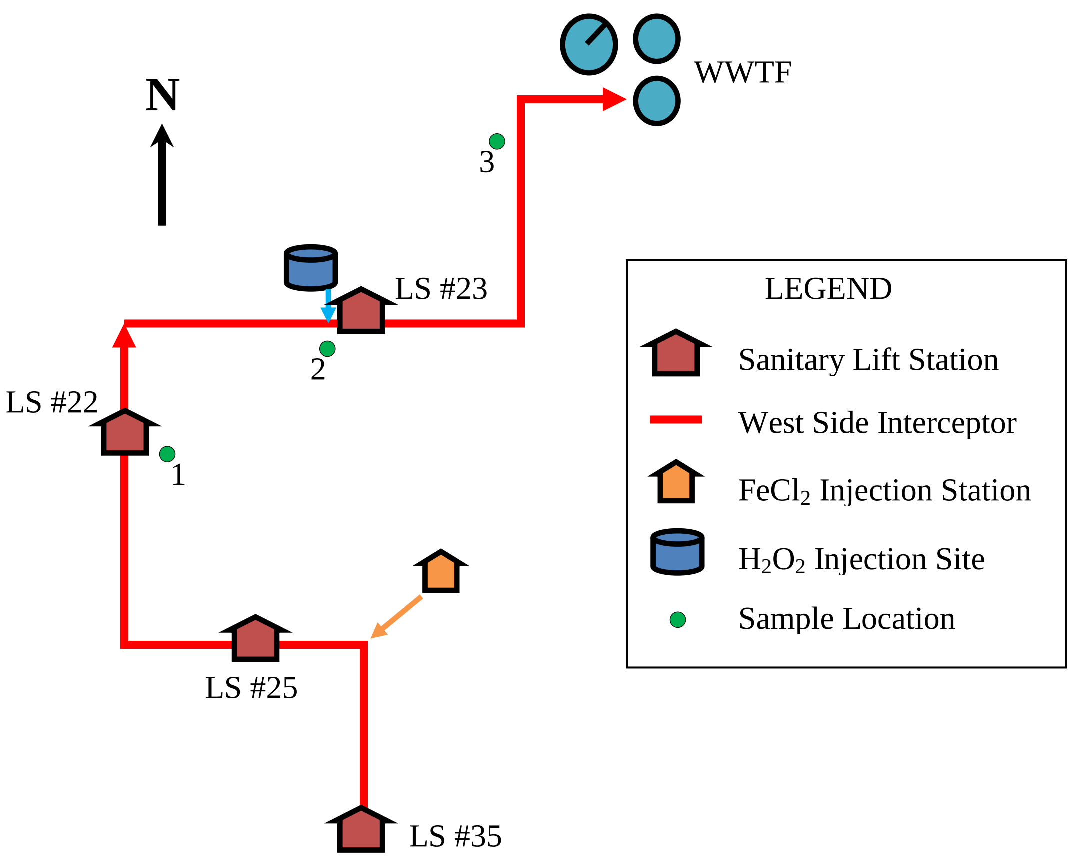

Figure 1. PRI-SC Demonstration Flow Diagram.

The sample locations (SL) used during the study were selected based on elevated H_2S levels previously recorded at these sites. A sudden directional shift in wastewater flow occurs at these locations resulting in turbulent conditions. SL1 and SL2 are sanitary lift station wetwells, and SL3 is a sanitary manhole. The demonstration was conducted in the summer and early fall to assess the PRI-SC process during the City's highest odor conditions.

Sample Collection

Sampling and monitoring parameters included measurement of wastewater temperature, pH, total and dissolved sulfides, atmospheric H_2S, as well as iron and H_2O_2 concentrations. Liquid grab samples were generally collected on a weekly rotational basis, three to four days per week, two to three times per day at each sampling location. H_2S monitoring was performed on a continuous basis via Odalogs at each of the sampling locations. The sampling parameters and methods are identified in Table 1 below.

Table 1. Sampling Parameters and Methods.

Liquid Grab Samples	**Procedure**
Total Sulfides	Std. Methods 4500-S^{2-} D. Methylene Blue (Lamotte drop count kit)
Dissolved Sulfides	Std. Methods 4500-S^{2-} D. Methylene Blue (Lamotte drop count kit) Using pre-flocculation to remove insoluble sulfides
pH	Combination glass electrode
Temperature	NIST calibrated thermometer
Total Iron	Std. Methods 3500-Fe D. Phenanthroline (Hach colorimeter)
Ferrous Iron	Std. Methods 3500-Fe D. Phenanthroline (Hach colorimeter) Using mild acidification to dissociate FeS
Residual H_2O_2	DPD redox test strips (EM Quant)
Vapor Samples	**Procedure**
H_2S	App-Tek OdaLog (continuous monitoring)

Baseline sampling was conducted throughout the WSI. The demonstration program focused on detailed sampling in the interceptor's northern reaches where high levels of sulfides were previously recorded. The sampling program was conducted in four phases that included:

1. Baseline sampling during which historical $FeCl_2$ dosing strategies were utilized,
2. PRI-SC demonstration where varied dosing rates for both H_2O_2 and $FeCl_2$ were employed,

3. Post PRI-SC sampling where historical $FeCl_2$ dosing strategies were repeated, and
4. Elevated $FeCl_2$ dosing where $FeCl_2$ injection rates were increased from historical levels to reduce sulfides to acceptable levels.

Phase three was conducted to obtain additional baseline sulfide information due to wet weather conditions experienced in June and July (Phase 1) of 2007. Phase four was conducted for comparison to identify a $FeCl_2$ injection rate that would result in similar sulfide levels achieved during the PRI-SC demonstration.

RESULTS

Phase 1

Throughout the study period, wastewater pH was essentially neutral varying between 6.8 and 7.6, with an overall median value of 7.3. Phase 1 of the study was conducted during July of 2007 and was performed to quantify existing sulfide concentrations in the northern reaches of the WSI using only the historical dosing strategy. During this period, $FeCl_2$ was introduced to the collection system at the existing injection station. The historical, flat dose of 17.9 mL/s was applied and sampling was conducted as previously outlined. Phase 1 sampling results are shown in Table 2 below. The listed sample locations are illustrated in Figure 1.

Table 2. Phase 1 Sampling Results.

Parameter	Sample Location 1	Sample Location 2	Sample Location 3
Average Total Sulfides (mg/L)	2.6	2.7	2.0
Average Dissolved Sulfides (mg/L)	0.2	0.6	0.6
Average H_2S (ppm)	-[a]	10	7.0
Maximum H_2S (ppm)	-[a]	50	45
Average Water Temperature (°C)	18.6	18.4	18.0

[a] H_2S data was not gathered at Sample Location 1 during Phase 1.

Wet weather conditions and subsequently cooler than normal wastewater temperatures occurred during Phase 1 of study likely impacting the sampling results. At all points along the WSI the average DS concentrations were below 1.0 mg/L. However, H_2S levels were elevated at SL2 and SL3 possibly due to the turbulent nature of these sites.

Phase 2

Phase 2 was conducted during August of 2007. During Phase 2 the PRI-SC process was demonstrated on a full-scale level to regenerate spent $FeCl_2$ for DS treatment in the downstream reaches of the WSI (e.g., SL3). Varying dosing rates of H_2O_2 and $FeCl_2$ were used to optimize sulfide treatment and minimize chemical consumption. H_2O_2 was injected in a sanitary lift station wetwell (i.e., SL2) and $FeCl_2$ was introduced to the collection system at the existing injection station. SL2 was selected as the H_2O_2 injection point due to historical sampling results that indicated residual $FeCl_2$ concentrations were minimized as this point. H_2O_2 injection rates of 2.2 and 3.5 mL/s and reduced $FeCl_2$ feed rates of 10.5 and 12.6 mL/s were used during the PRI-SC demonstration. Phase 2 sampling results are shown in Table 3 below.

Table 3. Phase 2 Sampling Results.

Parameter	Sample Location 1	Sample Location 2	Sample Location 3
Average Total Sulfides (mg/L)	1.4	4.1	1.4
Average Dissolved Sulfides (mg/L)	0.3	1.8	0.4
Average H_2S (ppm)	47	26	7
Maximum H_2S (ppm)	185	101	40
Average Water Temperature (°C)	20.8	20.4	20.3

Little variation in treatment occurred between the two dosing rates indicating that the lower H_2O_2 rate was as effective for the given treatment strategy. The sulfide concentrations upstream and at SL2 were greater than those measured in Phase 1, and higher wastewater temperatures were also recorded. In fact, the highest H_2S levels measured throughout the study were recorded at SL1 and SL2 during Phase 2. The elevated H_2S was attributed to high temperatures and low precipitation during August of 2007. During the PRI-SC demonstration an average total sulfide removal rate of 66% was observed downstream of SL2. Average DS and H_2S following H_2O_2 injection were reduced by 78% and 73%, respectively. Throughout Phase 2, the DS concentrations downstream of SL2 were consistently maintained below 0.5 mg/L, thereby exceeding the demonstration goal of maintaining DS $\leq$ 1.0 mg/L in the collection system.

In addition to sulfide removal, the efficacy of the PRI-SC process was confirmed by increased Fe^{2+} concentrations, which rose from 8.2 to17.8 mg/L following H_2O_2 injection. Residual H_2O_2 concentrations at SL3 were non-detectable. This result was expected due to the rapid reaction and degradation of H_2O_2 in wastewater (WEF, 2004). The required $FeCl_2$ injection rate was also reduced to 12.6 mL/s representing a 30% decrease from the previous flat dosing approach. The 10.5 mL/s $FeCl_2$ dosing rate was determined to be insufficient as sulfide treatment was reduced upstream of SL2.

Phase 3
Due to the wet weather conditions and cooler wastewater temperatures recorded during Phase 1, Phase 3 was conducted to obtain baseline sampling results and evaluate the effectiveness of the current flat $FeCl_2$ approach. Phase 3 was conducted during September of 2007. $FeCl_2$ was introduced to the collection system at the existing injection station at the previous rate of 17.9 mL/s. H_2O_2 injection was suspended at SL2. The sampling results obtained during Phase 3 are presented in Table 4 below.

Table 4. Phase 3 Sampling Results.

Parameter	**Sample Location 1**	**Sample Location 2**	**Sample Location 3**
Average Total Sulfides (mg/L)	1.2	3.2	3.3
Average Dissolved Sulfides (mg/L)	0.3	1.5	1.3
Average H_2S (ppm)	11	12	17
Maximum H_2S (ppm)	39	60	70
Average Water Temperature (°C)	20.3	20.0	19.7

During Phase 3, DS concentrations in the downstream reaches of the WSI were consistently greater than 1.0 mg/L. Additionally H_2S values were higher than 10 ppm. The Phase 3 results were more consistent with previously recorded data, and therefore the lower sulfide levels measured in Phase 1 were attributed to wet weather and cooler conditions. The results of Phase 3 also showed that the current flat $FeCl_2$ approach was not effective at controlling odors and sulfides throughout the collection system. SL3 H_2S monitoring data, from Phase 2 and 3, shown in Figures 2 and 3 on the following page illustrate the improved odor control in the downstream reaches of the WSI via PRI-SC versus the flat $FeCl_2$ dosing strategy.

Phase 4
Phase 4 was the final stage of the demonstration period. The goal of Phase 4 was to increase the $FeCl_2$ injection rate at the existing injection station to such a level as to obtain similar sulfide concentrations in the northern reaches of the WSI as those measured during the PRI-SC phase (i.e., Phase 2). By identifying a $FeCl_2$ rate with similar treatment efficiency to PRI-SC, a side-by-side comparison of the strategies could be performed. Phase 4 was conducted during the first two weeks of October 2007.

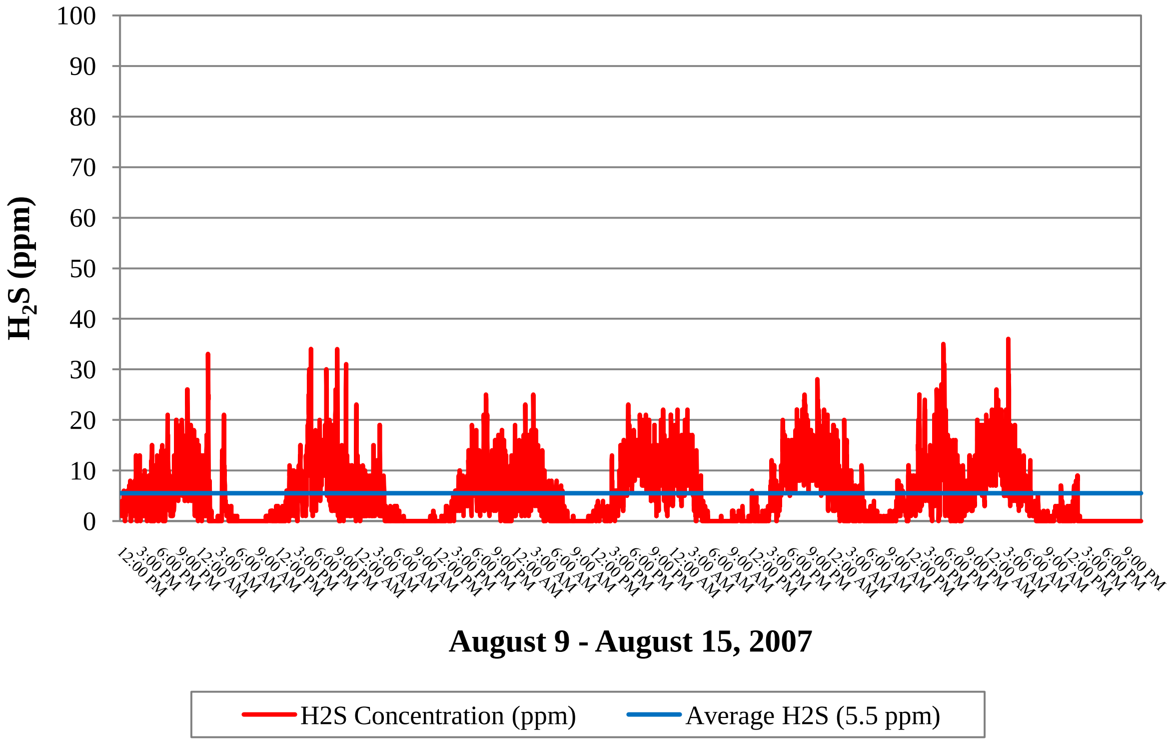

Figure 2. Sample Location 3 Hydrogen Sulfide Data – Phase 2.

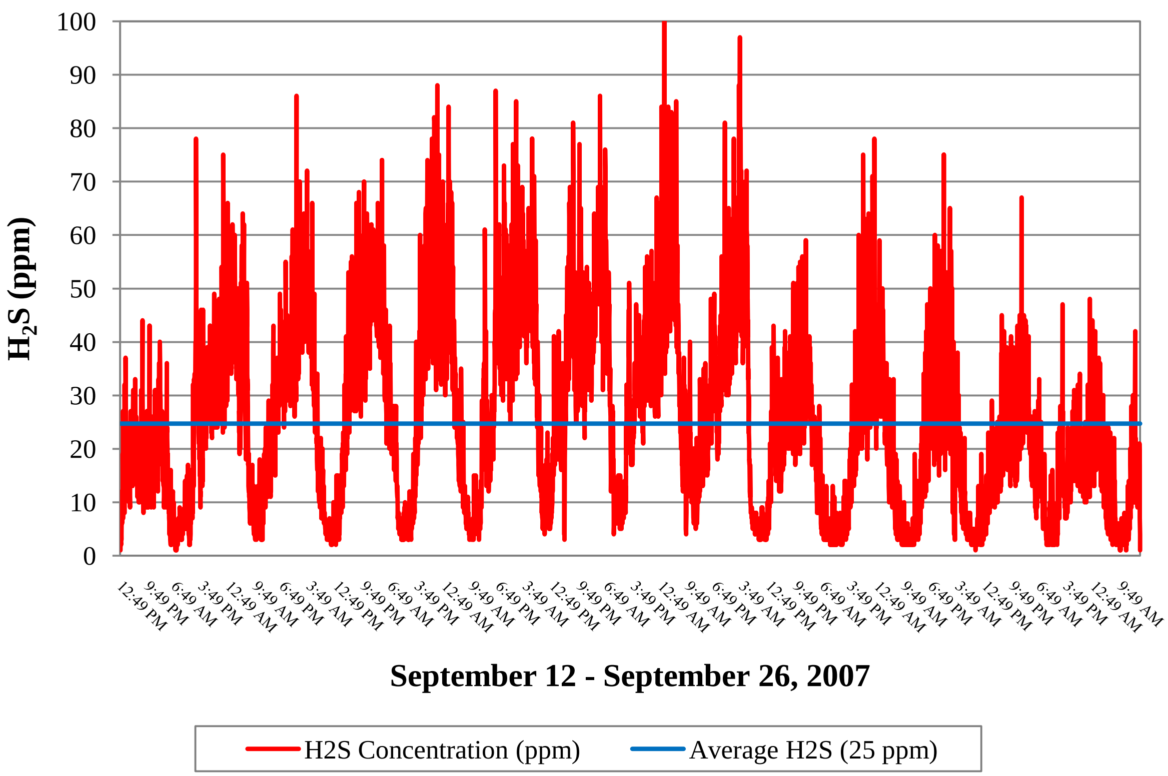

Figure 3. Sample Location 3 Hydrogen Sulfide Data – Phase 3.

Phase 4 sampling results are shown below in Table 5. To obtain similar sulfide levels in the northern reaches of the WSI, a $FeCl_2$ injection rate of 28.4 mL/s was required. The Phase 4 dosing represented a 59% increase from the previous $FeCl_2$ injection rate. Although the $FeCl_2$ rate was significantly increased, the iron alone strategy provided improved treatment throughout the WSI at all sample locations. Average H_2S and DS concentrations were consistently below 10 ppm and near 1.0 mg/L, respectively. It should be noted that the wastewater temperatures were lower in Phase 4 when compared to the PRI-SC period (i.e., Phase 2). Although not explored during the study, it is probable that the required $FeCl_2$ dosing rate may have been higher had the wastewater temperatures been analogous in Phases 2 and 4.

Table 5. Phase 4 Sampling Results.

Parameter	Sample Location 1	Sample Location 2	Sample Location 3
Average Total Sulfides (mg/L)	1.4	2.4	2.9
Average Dissolved Sulfides (mg/L)	0.3	1.1	1.0
Average H_2S (ppm)	2	4	8
Maximum H_2S (ppm)	22	40	40
Average Water Temperature (°C)	18.3	18.3	17.9

While improved sulfide control was realized through increased $FeCl_2$, the negative impacts associated with additional iron salt injection must be addressed. The 59% increase in $FeCl_2$ likely contributed to increased solids loading and sludge production at the WWTF. Though not investigated during this study, the overuse of iron salts can also result in reduced wastewater alkalinity (WEF, 2004). As previously stated H_2O_2 reacts in wastewater to form harmless byproducts and does not contribute to solids loading (USEPA, 1985).

To further evaluate the increased $FeCl_2$ approach and PRI-SC, an economic comparison was conducted based on the local 2007 market prices for $FeCl_2$ and H_2O_2. Though the treatment goal of DS $\leq$ 1.0 mg/L in the collection system was met using both an increased $FeCl_2$ approach and PRI-SC, treatment via PRI-SC resulted in a projected annual monetary savings ranging between $6,000 and $12,000 for the City of Fargo.

DISCUSSION

The flat $FeCl_2$ dosing strategy is no longer effective at controlling sulfides to acceptable levels throughout the collection system. Figures 4 and 5 below are data summary charts comparing the

Phase 2 and 3 total sulfides, dissolved sulfides and H_2S.

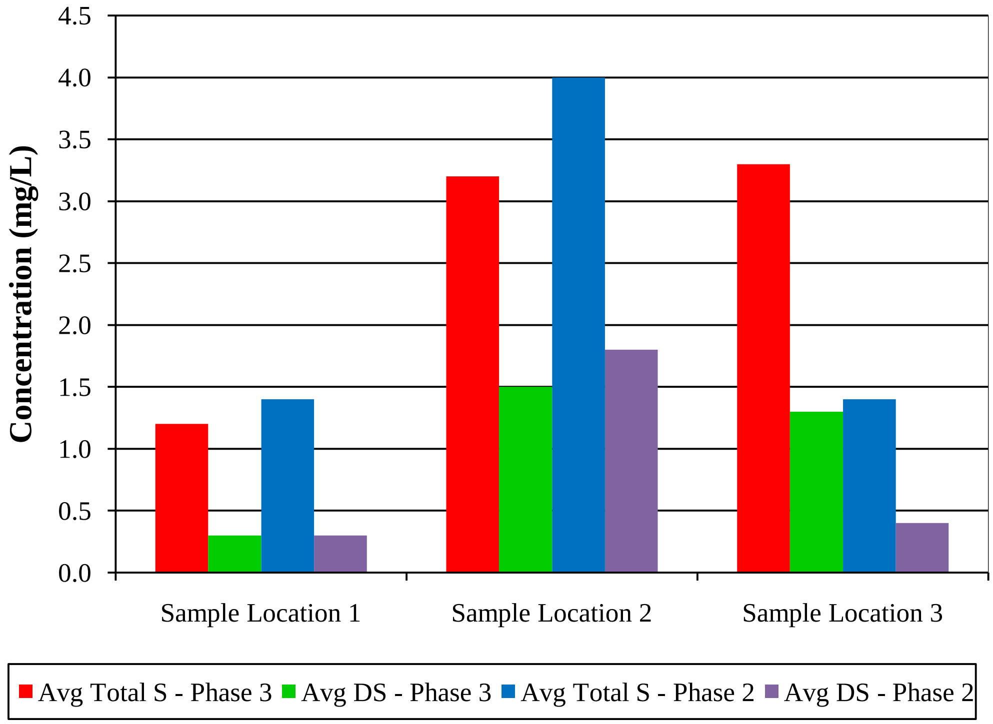

Figure 4. Phase 2 and 3 Average Total and Dissolved Sulfides.

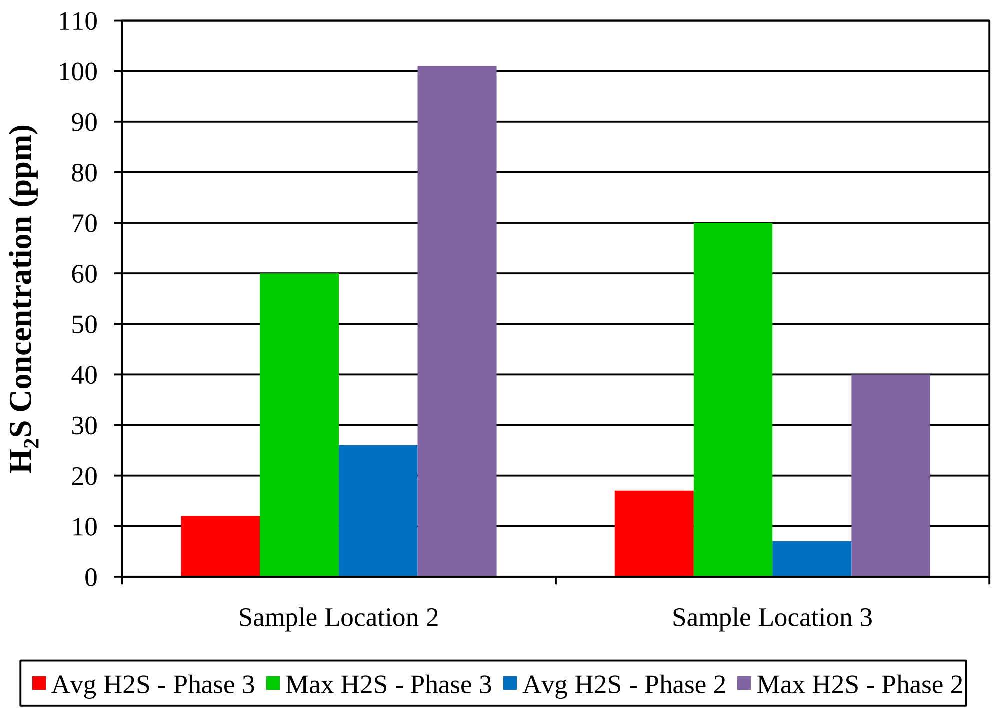

Figure 5. Phase 2 and 3 Average and Maximum Hydrogen Sulfide.

When compared to $FeCl_2$ treatment alone, the results of the demonstration program verified that PRI-SC was able to consistently provide effective sulfide control to less than 0.5 mg/L DS and to less than 10 ppm average H_2S downstream of the H_2O_2 injection point. Additionally, PRI-SC was able to reduce average DS concentrations downstream of the H_2O_2 injection site by 78%. Moreover, a 73% decrease in average H_2S was observed following H_2O_2 addition.

While the increased $FeCl_2$ approach analyzed in Phase 4 successfully met the study goal (DS ≤ 1.0 mg/L) increased iron salt addition may lead to unwanted side effects such as increased solids loading and alkalinity reduction. Additionally, the sulfide treatment levels achieved via PRI-SC (DS < 0.5 mg/L) were not attainable through elevated $FeCl_2$ injection.

In addition to treatment benefits, the efficacy of the PRI-SC process was confirmed by increased Fe^{2+} concentrations, which rose from 8.2 to17.8 mg/L following H_2O_2 injection. Furthermore, the amount of $FeCl_2$ necessary for treatment was reduced by 30% via the injection of H_2O_2 (thereby reducing solids loading to the WWTF headworks). An economic comparison was also conducted to analyze the costs of increased $FeCl_2$ dosing versus full-scale PRI-SC implementation for total sulfide control. By means of PRI-SC, an annual monetary savings ranging between \$6,000 and \$12,000 was achievable for the City of Fargo.

Although the monetary benefits of PRI-SC were minimal, the rising costs of iron salts may make the PRI-SC technology more economically advantageous in the future. Alternatively, the use of the proprietary PRI-SC process eliminates competition from other hydrogen peroxide suppliers. While the treatment benefits of PRI-SC are undoubtedly valuable to the City of Fargo, over time the costs associated with the continued use of the technology must be monitored as other alternatives for sulfide control may become more affordable.

CONCLUSIONS

In response to improved sulfide treatment, reduced $FeCl_2$ injection rates, and economic benefits realized during the demonstration program, the City of Fargo elected to incorporate PRI-SC as part of their upcoming collection system improvements.

Design of the proposed PRI-SC system was completed in the fall of 2008. Variable dosing strategies for $FeCl_2$ and H_2O_2 were incorporated for efficient odor control. The injection rates will fluctuate based on diurnal and seasonal sulfide loadings to prevent overdosing and reduce overall chemical costs. Hydrogen peroxide injection will be utilized to regenerate spent $FeCl_2$ at a new 151,410 m^3/d (40 mgd) sanitary lift station located in north Fargo. The PRI-SC technology will serve to reduce odors and corrosion in the 750 mm (30 inch) diameter forcemain from the new lift station and at the forcemain discharge at the WWTF headworks. The full-scale PRI-SC system will be placed into operation in the spring of 2010. Additionally, PRI-SC has been incorporated into the design of a new 124,915 m^3/d (33 mgd) sanitary lift station located in south Fargo. At a future date, full-scale PRI-SC will be implemented at this lift station when increased sulfide loading makes additional $FeCl_2$ injection impractical and uneconomical.

While PRI-SC was found to be beneficial for the City of Fargo, any liquid phase technology should be evaluated for effectiveness based on site specific conditions. Additionally, chemical costs fluctuate geographically and consideration must be given for variable market prices.

ACKNOWLEDGMENTS

Credits
The authors wish to acknowledge the staff of the Fargo Wastewater Treatment Facility for collaboration and assistance during the demonstration period. US Peroxide, LLC, is acknowledged for supplying reference materials related to their patented PRI-SC process. Peroxide Regenerated Iron - Sulfide Control (PRI-SC) is a registered trademark of US Peroxide, LLC.

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