PHARMACEUTICALS AND PERSONAL CARE PRODUCTS IN GROUND WATER FROM MUNICIPAL LAGOON TREATMENT

By

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A thesis

submitted in partial fulfillment

of the requirements for the degree of

Master of Science in Civil Engineering

Boise State University

December 2010

www.manaraa.com

BOISE STATE UNIVERSITY GRADUATE COLLEGE

DEFENSE COMMITTEE AND FINAL READING APPROVALS

of the thesis submitted by

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Thesis Title: Pharmaceuticals and Personal Care Products in Ground Water from Municipal Lagoon Treatment

Date of Final Oral Examination: 15 October 2010

The following individuals read and discussed the thesis submitted by student Teresa Nicholas, and they evaluated her presentation and response to questions during the final oral examination. They found that the student passed the final oral examination.

The final reading approval of the thesis was granted by George A. Murgel, Ph.D., P.E., Chair of the Supervisory Committee. The thesis was approved for the Graduate College by John R. Pelton, Ph.D., Dean of the Graduate College.

ACKNOWLEDGEMENTS

I wish to express my sincere thanks to the BSU thesis committee Dr. George A. Murgel, Dr. Sondra M. Miller, and Dr. James P. McNamara. Dr. Murgel, thank you for being my advisor and leading the thesis, which turned out to be a cross-discipline project. Dr. McNamara, thank you for your words of support, encouragement, and instruction. Thank you to Dr. Miller for joining the thesis committee so late in the process and providing reviews.

Thank you to the Idaho Department of Environmental Quality (DEQ) for funding the thesis project sampling. In addition, I wish to express my sincere thanks to Joe Baldwin, Idaho State Senior Environmental Hydrologist, for the guidance and sampling assistance with this entire thesis. This thesis would not have been possible without his support. Thank you to Ed Hagan, Idaho State Senior Ground Water Hydrologist, for encouragement and support through the DEQ ground water program. Thank you to Jeff Fromm, Idaho State Toxicologist, for the advice and partnership in presenting this thesis at multiple venues.

Thank you to Ed Furlong for your encouragement and results discussion after the sampling. Thank you to Shane Snyder who enlightened me on the practical aspects of the issues of PPCPs to drinking water systems. Thank you to George Tchobanoglosh for your time spent with me discussing the future issues of emerging contaminants and wastewater treatment technologies.

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Most importantly, thank you to Emily Nicholas, who missed her mother for the past three years on weekends and weeknights while I was working on this thesis. Thank you to my husband Gregg Nicholas for the financial support and family sacrifices made in order to complete the research and the thesis.

ABSTRACT

Pharmaceuticals and personal care products (PPCPs) are biologically active, organic chemicals that are introduced into the environment through wastewater streams. These chemicals are being found in the environment in trace concentrations and are of concern due to their unknown potential for harm to the health and welfare of the environment. The hypothesis is that during the course of wastewater treatment in a municipal lagoon system from a small community, PPCPs are seeping into ground water resulting in chronic low exposure in the environment. The objective of this work was to study a specific wastewater lagoon treatment system in Mountain Home, Idaho and the surrounding ground water for PPCPs. Water samples were taken from influent, lagoons, and surrounding ground water wells to look for presence of PPCPs. A conceptual model of the ground water flow was developed in order to link lagoon seepage to surrounding ground water wells. The ground water flow model combined with the sampling data was used to show that PPCPs are present in the lagoons and seeping into the ground water in very low yet detectable concentrations. PPCPs were detected in the samples from the headworks, in the storage lagoon, and in a monitoring well downgradient of the lagoon. Twelve PPCPs were tested and eleven were found in the headworks in concentrations up to 7,920 ng/L. Seven PPCPs were found in the storage lagoon in concentrations up to 880 ng/L. Six PPCPs were found in a downgradient monitoring well in concentrations up to 82 ng/L.

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INTRODUCTION AND HYPOTHESIS

Pharmaceuticals and personal care products (PPCPs) are biologically active, organic chemicals found in the environment at trace concentrations (Daughton 2004; Kummerer 2004; Heberer 2002; Ternes and Joss 2006; EPA 2008; USGS 2002). Due to their unknown potential for carcinogenicity, biological activity, and promotion of antibiotic resistance in certain organisms, PPCPs are an emerging concern (Daughton 2004; EPA 2008; USGS 2002). PPCPs are introduced into wastewater streams through household drains and subsequently enter municipal wastewater treatment systems (EPA 2008; Richards 1996; Bound and Voulvoulis 2005). Subsequently, wastewater enters the environment through seepage from all types of treatment facilities or direct discharge of treated water to the environment (EPA 2008; Kummerer 2004; Ternes and Joss 2006). This transmission of water from household to treatment system to ground water represents a major potential pathway for PPCPs to enter the environment (EPA 2008; Dingman 2002; Domenico and Schwartz 1998; USGS 2008a).

While a number of studies have been performed on large municipal treatment systems to understand the interactions of PPCPs with the environment, little work has been performed to investigate the lagoon style wastewater treatment systems that are prevalent throughout the United States (EPA 2002). It is possible that PPCPs are seeping into ground water from the wastewater treatment lagoons or land application of treated wastewater; however, studies to date linking PPCPs and lagoon seepage are limited

(EPA 2007b). It has been hypothesized for this study that during the course of wastewater treatment in a lagoon system and subsequent land application of the effluent, some PPCPs are seeping into ground water in a form or concentration that can be detected using currently available analytical techniques. In addition, wastewater treatment lagoons and land application of wastewater form part of the exposure route of trace chemicals in the environment. Grab samples of wastewater and area ground water in the vicinity of a selected lagoon system have been collected and evaluated for both presence and trend analysis of PPCP concentrations as they relate to the site-specific conditions. The results will provide information about possible exposure routes of trace chemicals in the environment from wastewater treatment lagoons.

Scope of Thesis

There are approximately 200 wastewater land application sites permitted by the Department of Environmental Quality (DEQ) throughout the state of Idaho; these wastewater reuse permits were examined to identify a suitable project site that used facultative lagoons as the wastewater treatment process. Once identified, the site was researched for historical information on wastewater treatment (flow, lagoon specification, detention time, and land application), population base, and site geology (soils, vadose zone, aquifer systems, ground water recharge, ground water flow direction, and existing wells). The presence and concentrations of PPCPs are known to be influenced by economic and demographic structure (Trapp and Matthies 1998) and thus were important criteria in the site selection. Water samples were collected before and after wastewater

treatment, as well as from ground water. Sample sites included a municipal wastewater lagoon and water bodies downgradient from wastewater treatment. Samples were analyzed for PPCPs and changes in PPCP concentration from influent to effluent to the local ground water system and were evaluated to develop a conceptual model for fate and transport of PPCPs. Oxygen and deuterium isotope data were used to determine linkages between treatment system sources of the chemicals and the concentrations found in the local and regional ground water. These data, along with concentration gradients, were used to establish hydraulic connectivity between the wastewater lagoon seepage and ground water. A qualitative analysis was performed, excluding a quantitative analysis of the transport of the PPCPs from the lagoon due to the limited budget and to limit the scope of research.

Background

Environmental Pathways for Pharmaceuticals and Personal Care Products

Trace chemicals that are biologically active in humans and other vertebrates are found around the world in very low concentrations in ground water, rivers, lakes and coastal waters (EPA 2008; Kummerer 2004; Ternes and Joss 2006). This thesis will focus on a subset of these contaminants that includes both pharmaceuticals and personal care products (PPCPs). As shown in [Figure 1,](#page-13-0) PPCPs are introduced into wastewater streams through household and hospital drains and subsequently enter municipal wastewater treatment systems (Richards 1996; Metcalf and Eddy 1991; Bound and Voulvoulis 2005).

Figure 1 Pathways for PPCPs (adapted from EPA 2008; Kummerer 2004; USGS 2002; Ternes and Joss 2006; Garg and Jha 2005; Heberer 2002)

Many current wastewater treatment methodologies were not designed to remove PPCPs (EPA 2007b; Heberer 2002; Gobel et al. 2007; EPA 2008). Some studies indicate that secondary wastewater treatment processes (i.e., activated sludge and membrane bioreactors) only partially remove PPCPs (EPA 2007b; Heberer 2002; Gobel et al. 2007; EPA 2008). Studies indicate that post-treatment effluents from these processes contain PPCPs (EPA 2008; USGS 2008b; Snyder et al. 2004). Information is more limited on the fate and transport of PPCPs from facultative treatment lagoons. Because there are over 7,000 facultative lagoons in use in the United States located in less populated cities and towns (EPA 2002), their design and use has led to wastewaters entering the environment through seepage from the treatment facilities or direct discharge of treated water to the environment (EPA 2008; USGS 2008b). The cycling of water from household to

treatment system to ground water represents a major potential pathway for PPCPs to enter the environment (Heatwole and McCray 2007; Garg and Jha 2005; Hai-long and Zu-xin 2006; Connell and Van den Dael 2003; Marshall et al. Jaffe, 2000; USGS 2007). However, limited information now exists on the fate of these chemicals in soils and ground water from any treatment source, especially from wastewater lagoon systems (EPA 2008; USGS 2008b). In order to explore the environmental fate and transport of PPCPs, this study considered the topics of pharmacodynamics, wastewater treatment by facultative lagoons, ground water hydrology, ground water chemistry and stable isotopes for identification of respective PPCPs.

PPCPs enter wastewater in three primary ways. In urine and feces, humans excrete small quantities of the pharmaceuticals they consume (EPA 2008; Drug Information Online 2008). While the metabolism of pharmaceuticals varies by individual, the principal means of elimination is generally through the urine stream (Hughes 1996; Drug Information Online 2008; EPA 2008). Additionally, unused pharmaceuticals enter wastewater when flushed into the sewer system, as has commonly been recommended as a means of disposal (EPA 2008). Finally, personal care products, such as shampoo and soap, enter wastewater when they are washed off any part of the body (EPA 2008). The following section describes wastewater treatment processes involving municipal wastewater lagoons and land application of treated wastewater, both of which are possible sources of PPCPs to the environment.

Wastewater Treatment

Facultative Lagoons

Wastewater treatment methods impact the amount of and potential for specific PPCPs to be present in effluent (EPA 2008; Heberer 2002; Conn et al. 2006; Matamoros et al. 2005). PPCPs are organic compounds and to date, limited data exist on the fate of PPCPs in facultative lagoons (EPA 2008; EPA 2007b). The most important removal pathways of organic compounds during wastewater treatment are: biotransformation/biodegradation, adsorption by the sludge (excess sludge removal), and stripping by aeration (volatilization) (Richards 1996; Metcalf and Eddy 1991; Qasim 1999). Lagoons have primarily been studied as a source for nitrogen loading to ground water (DeSutter et al. 2005). Knowledge of how facultative lagoons work is fundamental for understanding the potential fate of PPCPs in this thesis.

Facultative lagoons are ponds designed to hold and treat wastewater through a combination of physical, biological (aerobic and anaerobic degradation reactions), and chemical processes [\(Figure 2\)](#page-16-0) (EPA 2002; Harris 2003). Each zone has the potential to degrade organic compounds, including PPCPs. Lagoons can remove settleable solids, biochemical oxygen demand, pathogens, fecal coliform, and ammonia (EPA 2002). Lagoons are a treatment system relying on complex biochemistry to degrade organic wastes present in wastewater as contained in an earthen berm constructed tank environment (EPA 2002; Qasim 1999; Richards 1996; Metcalf and Eddy 1991). Detention time allows solids to settle and aerobic, facultative and anaerobic zones are created in the ponds (EPA 2002; Qasim 1999; Richards 1996; Metcalf and Eddy 1991).

As [Figure 2](#page-16-0) shows, each of these zones is biologically and physically active and different chemical processes treat the wastewater (Harris 2003).

Figure 2 Wastewater Treatment Zones in a Facultative Lagoon (adapted from Metcalf and Eddy 1991; Harris 2003)

Lagoon systems are prevalent in use in the United States (EPA 2002), and there are more than 400 lagoon treatment systems in Idaho according to the Idaho Rural Water Association (2008). The simple design, operations, and low capital costs make them a cost effective wastewater treatment method (Army Corps of Engineers; Muga and Mihelcic 2008; Zhang 2001). Compared to mechanical wastewater treatment systems, lagoon systems can cost four to five and a half times less (Muga and Mihelcic 2008), which makes them a viable option for many communities, and socio-economic

considerations. Lagoon systems also have the potential to contribute PPCPs to the environment, making this current study of them timely and important.

Lagoons seep liquids, which percolate into the subsurface. This thesis considers wastewater seepage from lagoons as a potential path for PPCPs to enter into the environment. Lagoon seepage could provide a major source of PPCPs to enter into the environment (see [Figure 3\)](#page-17-0).

Figure 3 Lagoon Seepage Into Ground Water (adapted from Dingman 2002; Freeze and Cherry 1979)

The current Idaho Wastewater Rules (IDAPA 58.01.16.493) specify design

standards for a maximum seepage rate of five hundred gallons per acre per day for new

lagoon construction. In contrast to the design standards, the rules also require seepage testing to show that existing lagoons constructed after April 21, 2007 seep less than 0.125 inches (3400 gallons) per acre per day. Existing lagoons constructed before April 21, 2007 may seep up to 0.250 inches (6800 gallons) per acre per day. Lagoons must be tested every ten years for seepage to ensure compliance with the rules. After evaluating lagoon seepage testing reports from 2005 through 2008 (DEQ 2009), the results indicated that all municipal lagoons tested to date seep to some extent. Lagoons may be designed to have minimal seepage, but during the course of use operational and maintenance issues (such as membrane punctures, weeds, slope stability, or cleaning) can create conditions where seepage occurs or increases (Harris 2003). Nitrates and ammonia are two possible drinking water contaminants that can seep from lagoons into ground water. The EPA has set maximum contaminants levels (MCL) for nitrates and ammonia in drinking water, which in turn limits how much of each parameter may seep from a lagoon. The EPA has not, however, established MCLs for the PPCPs under consideration in this thesis, leaving PPCPs currently unregulated (EPA 2008; Idaho Department of Administration 2008).

Wastewater Land Application

After lagoon treatment, wastewater effluent may still contain PPCPs and, if land applied, could contribute additional PPCPs to ground water after percolating through the soils. Applying wastewater to land is a way to naturally treat wastewater through soil filtration, biological activity, and plant uptake of nutrients; provide aquifer recharge; and, eliminate the need for wastewater discharge directly into a surface water body (DEQ 2007; Heatwole and McCray 2007). Land application of treated wastewater allows for

water reuse, as it can provide a source of irrigation water and nutrients for crops (DEQ 2007; Coppola et al. 2004). A natural treatment system, wastewater land application is generally constituent limited (i.e., hydraulics, nutrients, salts, etc) and considers agronomic rates of nutrient uptake when crops are grown using the wastewater (DEQ 2007). Wastewater land application is managed to address where the water travels, its surface residence time, its subsurface flow paths, water quality impacts to ground water, and the amount of crop cover uptake for both water and its nutrients (DEQ 2007). Wastewater land application with a crop system, as seen in [Figure 4,](#page-19-0) is considered a treatment system, not a disposal mechanism (DEQ 2007).

Figure 4 Potential Treatment of Wastewater from Land Surface to Ground Water (adapted from DEQ 2007)

Because land application of wastewater is a treatment system, the additional treatment processes may be important when evaluating the PPCP contribution in the land applied water. In addition to the typical wastewater processing in the soil layers, there are potential PPCP-specific processing mechanisms that are shown in [Figure 5.](#page-20-0)

Figure 5 Potential for PPCPs Treatment in Subsurface (adapted from EPA 2008; Hai-long and Zu-xin 2006; Muller et al. 2007; Kummerer 2004)

The goal is to apply wastewater at a rate and manner that will allow the sitespecific crop system to assimilate the wastewater constituents such that minimal amounts will leave the site through leaching or runoff (DEQ 2007). Wastewater land application and treatment may be analogous to a controlled precipitation event because the goal is to apply wastewater at a rate and manner for optimizing crop uptake of constituents

(DEQ 2007). Wastewater land application is a seasonal land use system (DEQ 2007) that relies on site-specific design and operation to prevent hydraulic overloading, ground water contamination, and impacts to surface water (such as from runoff) at the application site (DEQ 2007; Hai-long and Zu-xin 2006). Parameters currently monitored at Idaho wastewater re-use sites are site- and system-specific for system design and operation and include hydraulic and constituent loading (i.e., nitrogen, total dissolved solids, phosphorus, and chemical oxygen demand) (DEQ 2007). The fate and transport of PPCPs is not regulated and thus not monitored in wastewater treatment lagoons, wastewater reuse sites, or subsequently in the receiving ground water (EPA 2008; Idaho Department of Administration 2008). It is now being recognized that, after treatment, wastewater still contains trace PPCPs that are biologically active (EPA 2008; USGS 2002; USGS 2008a; Drewes 2007; Snyder et al. 2004). The fate and transport of these emerging contaminants involves a variety of complex processes, and further understanding of the potential pathways is needed (EPA 2008; USGS 2002; USGS 2008a; Snyder et al. 2004).

Environmental Pathways and Fate of PPCPs in Wastewater

Throughout the wastewater treatment process, air, water, soil, and biota present multiple potential pathways for PPCPs present in wastewater to enter the environment (Metcalf and Eddy 1991; Qasim 1999; Richards 1996; Fetter 1999). Three factors influence fate and transport of PPCPs: 1) the unique physiochemical properties of PPCPs, 2) the transport properties of the receiving environment (see [Figure 2](#page-16-0) and 3), and 3) chemical transformations of PPCPs along the transport process, which are important in

interface dynamics (Domenico and Schwartz 1998; Kinney et al. 2006; Fetter 1999; Crosby 1998). In the dynamic and variable environment that PPCPs encounter during wastewater treatment, PPCPs partitioning coefficients may be important for predicting what will occur during vapor/solid, vapor/liquid, liquid/liquid, or liquid/solid stages in treatment and in the environment (Kummerer 2004; Fetter 1999; Crosby 1998). The tendency of PPCPs to be in water versus sorbed can provide information about where it might exist in the wastewater treatment system. The octanol-water distribution coefficient (K_{ow}) will "indicate the tendency of an organic chemical to partition to lipids or fats, sorb to particulates such as soils, sorb to biomass or sludge or distribute among the various environmental compartments" (Kummerer 2004; Fetter 1999). For example, sorption affinity will be influenced by the molecule's K_{ow} , its aqueous solubility and its molecular structure (Kinney et al. 2006; Kummerer 2004). The less polar a chemical or PPCP is the more potential for hydrophobic partitioning to organic matter to occur (Kinney et al. 2006). The greater the hydrophobicity of the chemical, the greater will be the tendency for the PPCPs to partition into the hydrophobic organic phase (Kinney et al. 2006). The greater the chemical's K_{ow} , the greater will be the tendency for low water solubility, a large soil/sediment adsorption coefficient, and a large retardation factor (Kinney et al. 2006; Lissemore et al. 2006; Kummerer 2004). A flow path relationship has been shown to exist with PPCP concentrations (Lissemore et al. 2006). Past studies indicate that water-soluble chemicals follow water flow paths and chemicals with high K_{ow} values tend to be found in soils with high total dissolved organic carbon (Lissemore et al. 2006). These trends suggest that hydrophilic PPCPs may not attenuate during lagoon treatment.

Therefore, understanding a PPCP's known chemistry with particular emphasis on K_{ow} will provide clues for defining pathways to degradation, sorption, or transport. A sorption guide has been proposed (Drewes 2007; Rogers 1996) that refers to a chemical"s octonal water partitioning coefficient where a $log K_{ow}$ less than 2.5 will have low sorption potential (Drewes 2007; Rogers 1996). A log K_{ow} between 2.5 and 4 will have medium sorption potential and a $\log K_{ow}$ greater than 4 will have a high sorption potential (Drewes 2007; Rogers 1996).

In evaluating wastewater treatment systems and PPCPs, the $log K_{ow}$ provides predictive evidence for understanding the pathway a PPCP will take, i.e. water or soil (sludge). For PPCPs in wastewater, it has been suggested that the concentration difference between the influent and effluent provide the clearest indicator of total removal efficiency when based upon a mass balance change (Aga 2008).

PPCP Pharmacodynamics

Understanding PPCP pharmacodynamics, the reactions between drugs and living systems, is needed for proper evaluation of the sampling results. The chosen PPCPs were designed to be biologically active chemicals that target end users, so that their direct effects, which include their mode of action and side effects, are primarily understood (Drug Information Online 2008). The chemistry of the chosen PPCPs is variable and little is known about chronic low dose exposure, effects on non-target organisms, and/or environmental fate and transport (Aga 2008; Drug Information Online 2008; Crosby 1998; Jjemba 2008; Snyder et al. 2004). As a group, a homogeneous set of

characteristics both in vivo and in the environment is not expected (Aga 2008; Drug Information Online 2008; Crosby 1998; Jjemba 2008; Bateman 2001). Pharmaceutical metabolism generally occurs in vivo and involves chemical changes that most often convert PPCPs into more readily excreted polar products, thus making them water soluble (Kummerer 2004; Jjemba 2008; Bateman 2001; Snyder et al. 2004). In general, most drugs are lipophilic chemicals (Nowak 2005) and their metabolites become more hydrophilic for elimination (Kummerer 2004; Drug Information Online 2008). This is an important characteristic in the discussion on environmental fate and future study needs. Another general point is that pharmaceuticals and their metabolites in addition to being biologically active are often resistant to degradation. PPCPs may be excreted as the original "parent" compound, as a conjugate, as metabolite(s) or a combination of the three (Aga 2008; Crosby 1998; Kummerer 2004; EPA 2008; Jjemba 2008). The wastewater collection system as discussed earlier, collects PPCPs as they are washed off of the body, flushed down household drains, or passed out of the body through elimination. The target PPCPs for this study are in [Table](#page-24-0) 1 and the following discussion of carbamazepine, gemfibrozil, sulfamethoxazole, and estrogens is needed for further understanding of this study.

PPCPs	Use	Molecular Weight	Log $[K_{ow}]$
Caffeine	Stimulant	194.2	-0.07
Carbamazepine	Anti-seizure	236.3	2.45
Esterone	Estrogen	270.4	3.13

Table 1 PPCP Physiochemical Properties (adapted from Snyder et al. 2004)

Carbamazepine

Carbamazepine, an anti-psychotic, anti-epileptic, anti-neuralgic, and anti-diuretic drug (Drug Information Online 2008; RxList 2009), is one of the most frequently detected PPCPs in surface waters and not naturally present in ground water (Clara et al. 2005; Miao et al. 2005; Metcalfe et al. 2004; Godfrey et al. 2007; Gagne et al. 2006). Carbamazepine is a highly polar molecule, (Aga 2008; Daughton 2007; Drewes 2007; Jos et al. 2003), about 3% is excreted unchanged in the urine (Daughton 2007; Drewes 2007; Jos et al. 2003) and limited studies indicate that less than 10% is removed during sewage treatment (Ternes et al. 2004; Hernando Guil et al. 2007; Miao et al. 2005). Carbamazepine has been found in ground water with no degradation shown in travel times of more than 6 years (Heberer 2002; Godfrey et al. 2007; Aga 2008; Snyder et al. 2004). These characteristics have led to speculation that carbamazepine may be considered as an ideal tracer and could serve as an indicator of wastewater seepage (Clara et al. 2004; Haack et al. 2009). Ideal tracers are chemicals where there is no sorption or reactions that will occur with it in the system of study (Dingman 2002; Trapp

and Matthies 1998). Ideal tracers can be a chemical that is present at measureable levels, and is not attenuated in the vadose zone or impacted by vegetation (Dingman 2002).

Sulfamethoxazole

Sulfamethoxazole is a sulfonamide-based drug. Sulfonamides are generally used as antibacterial agents and are the most used class of antimicrobials in the world for humans and livestock (Kummerer 2004; Metcalfe et al. 2004). Their mode of action is through competitive inhibition of bacterial folate biosynthesis, which is needed for nucleic acid synthesis and thus cellular division (Ternes and Joss 2006). This is a process that may have important implications to biological (aerobic and anaerobic) degradation reactions in facultative lagoons. The overall biodegradation in wastewater treatment may be slowed down by antibiotics deactivating the microbial degraders. Since antibiotics are included in the wastewater mixtures, there may be plant operation and treatment issues that occur as a result of deactivating microbial degraders in treatment (Jjemba 2008). This could be important to the discussion of the results if antibiotics are found in the lagoons. Additionally, as antibiotic resistant bacteria can be found in wastewater (Volkman et al. 2004), the antibiotics found in wastewaters may have a potential role in the spread and maintenance of multi-resistance of bacterial pathogens (Ternes and Joss 2006; Boreen et al. 2004; Gobel et al. 2005).

Sulfonamides are generally metabolized by acetylation in humans and only about 15-20% of the active drug appears in the urine (Aga 2008; Ternes and Joss 2006; Gobel et al. 2005). Sulfonamide residues are potentially carcinogenetic, and one form, sulfamethazine, is a thyroid carcinogen (Metcalfe et al. 2004), necessitating the

evaluation of concentrations in wastewaters as an important factor for risk assessment in future studies. During wastewater treatment, there is potential for retransformation back to the parent compound (Gobel et al. 2007; Gobel et al. 2005). Sulfonamide removal in activated sludge and membrane bioreactor (MBR) treatment has been shown to be variable but overall incomplete so that it is detectable in the effluent (Karthikeyan and Meyer 2006; Gobel et al. 2007). Secondary treatment methods generally reduce sulfonamides via sorption and transformation processes (Karthikeyan and Meyer 2006; Gobel et al. 2007). Sulfamethoxazole is hydrophilic (refer to [Table](#page-24-0) 1) and the mostly commonly detected form in a study looking at sulfonamides (Gobel et al. 2005). Sulfamethoxazole has been found in ground water samples (Focazio et al. 2004) and in shallow ground water wells hydraulically downgradient from a community septic tank drainfield (Godfrey et al. 2007). When considering its environmental fate in a lagoon, a study on sulfamethoxazole showed that it may undergo slow photo-degradation in lakes (Boreen et al. 2004).

Gemfibrozil

Gemfibrozil is an antihyperlipidemic drug that reduces triglycerides and increases cholesterol carried in high density lipoprotein (Drug Information Online 2008). Gemfibrozil forms metabolites that are eliminated in urine and feces. Six percent of the dose can be accounted for in the feces and approximately seventy percent of the administered human dose is excreted in the urine, with less than 2% excreted as the unchanged parent compound (Drug Information Online 2008). Gemfibrozil has a high hydrophobicity (refer to [Table](#page-24-0) 1) and with this tendency to partition into the hydrophobic

organic phase, it has also been found in treated effluent from wastewater systems with removal efficiencies ranging from 6 to 50% (Quinn et al. 2008; Gagne et al. 2006; Daughten and Ternes 1999). Studies also suggest it may be one of the ten most abundant PPCPs found in wastewater effluent (Gagne et al. 2006). Studies with gemfibrozil indicate a concern for embryonic effects in mammals and that its toxicity rating should be reclassified (Quinn et al. 2008). This makes its environmental presence important for evaluation.

Estrogens

Estrogens occur in males and females during their entire lifetime and have different biological effects on the different target tissues (EPA 2008; Raftogianis et al. 2000; Okayasu et al. 2005). In addition to reproductive organs, estrogens act on the brain, bone, liver, and heart (Raftogianis et al. 2000; Okayasu et al. 2005). Estrogens are eliminated from the body by conjugation whereby a hydrophilic side chain is attached (glucuronic acid or sulfate) making it more soluble to enter the urine stream (Raftogianis et al. 2000; Okayasu et al. 2005; Johnson and Williams 2004). Studies suggest that de-conjugation can occur through enzymatic hydrolysis in the wastewater collection system or treatment process (Drewes 2007; Snyder et al. 2004; Okayasu et al. 2005; Johnson and Williams 2004). It is suggested that estrogens have high potential to be adsorbed to sewage sludges and that longer solids retention times may result in better removal efficiency of estrogens (Drewes 2007). This may be important at the study site with a 22 day detention time traveling through the lagoons and an even longer detention in the storage lagoon. It has been shown that there is a 60 to 90% removal efficiently of

estrogens from wastewater using MBRs and conventional activated sludge wastewater treatment methods (Hernando Guil et al. 2007). The bioavailability of estrogens to influence the environment makes estrogens an important choice for evaluation in wastewaters (Aga 2008; Clara et al. 2005; Conn et al. 2006; Drewes 2007; Snyder et al. 2004; Daughten and Ternes 1999; Jos et al. 2003; Daughton 2007; Gagne et al. 2006).

Nitrogen

Nitrogen is often used as an indicator for fecal contamination of ground water and is being studied to determine if there is a correlation between monitoring parameters and PPCPs (Jjemba 2008; Haack et al. 2009). As such, there may be a correlation between PPCPs and nitrogen transport through the vadose zone and into ground water. Many studies have shown that nitrogen transport from surface water sources into ground water occurs (EPA 2008; Heatwole and McCray 2007; Garg and Jha 2005; DeSutter et al. 2005; Haack et al. 2009). Elevated nitrogen concentrations in ground water have been implicated with wastewater treatment systems and agricultural operations (Heatwole and McCray 2007; Garg and Jha 2005) and shallow aquifers may be vulnerable to nitrate contamination (Garg and Jha 2005). Nitrogen removal from this wastewater treatment lagoon system may also correlate with some of the chosen PPCPs. If nitrogen is found in the ground water with PPCPs, the total removal efficiency of the PPCPs and nitrogen at each sampling location can be evaluated.

Oxygen and Deuterium Isotopes

Isotopes of oxygen and deuterium, a hydrogen isotope, have been shown to provide a line of evidence toward establishing hydraulic connectivity, between surface water and ground water (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005). The fractionation of the stable isotopes of oxygen (^{18}O) and deuterium (^{2}H) in atmospheric water vapor is subject to changes when water evaporates and condenses (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005). A linear correlation can be inferred as it relates to water that has not undergone evaporation (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005). Therefore, water with different evaporative histories can have unique isotopic fingerprints (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005). Oxygen and deuterium ratios can be plotted and compared to the Meteoric Water Line (MWL) (see [Figure 6\)](#page-31-0), which is the annual average isotope composition of precipitation at locations around the globe (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005). The relationship between ${}^{18}O$ and ${}^{2}H$ in meteoric waters is as follows: δ^2 H = 8 δ^{18} O +10‰ (SAHRA 2005).

Water that has been unaffected by physio-chemical processes (evaporation) should fall on the MWL line as it should contain more of the lighter oxygen, as seen in [Figure 6](#page-31-0) (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005).

Figure 6 Summary of How Hydrological Processes Affect Oxygen and Hydrogen Isotopic Composition of Water (SAHRA 2005)

As water becomes more depleted in the lighter isotopes through evaporation, it will deviate from the MWL (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005). The greater the evaporation the farther away the sample will be from the MWL (Domenico and Schwartz 1998; Freeze and Cherry 1979; SAHRA 2005). The same or similar isotopic signatures suggest hydraulic connectivity between surface water sources and ground waters (SAHRA 2005; Coplen et al. 2000). In this study, oxygen and deuterium isotope ratios are used to provide evidence to evaluate the connectivity of lagoon effluent seepage to ground water (SAHRA 2005; Coplen et al. 2000), because wastewater is subject to evaporative processes during its travel time through a lagoon (Metcalf and Eddy 1991). The same or similar isotopic signature found in lagoon effluent

and ground water may suggest that the effluent, which may contain PPCPs, is recharging ground water.

Study Location

In order to provide a basis for understanding PPCP contamination contributions from wastewater to ground water, a study location was needed that could be searched for pharmaceuticals in the environment, and assessed for hydrology with respect to movement of pharmaceuticals to the ground water. A specific study site was chosen from the Idaho Wastewater Reuse permitted sites list. The chosen study location was the City of Mountain Home Idaho due to its wastewater lagoons, existing monitoring wells and permitted wastewater land application site. The City of Mountain Home is located in Elmore County, in southwestern Idaho [\(Figure 7\)](#page-33-0).

The city had a population of 11,143 according to the 2000 census. The population has grown to more than 12,500 according to the 2004 estimated census. The 2000 census indicated that the population base is distributed in age with 29.6% under the age of 18, 9.8% between the age of 18-24, 32.8% between the age of 25-44, 18.1% between the age of 45-64, and 9.7% over the age of 65. The median age was 32 years (Mountain Home Economic Development 2004). This residential population contributes the PPCP loading to the wastewater stream.

Figure 7 Mountain Home Study Area Showing City of Mountain Home, and Wastewater Treatment Facility and Extent of Irrigated Agricultural Area East of Wastewater Treatment Facility (adapted from Baldwin et al. 2009)

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City of Mountain Home Wastewater Treatment

The wastewater treatment facility is located approximately 2 miles south of the city in Elmore County, in sections 1, 2, 11 and 12, Township 4S Range 6E (Keller Associates 2006). The location is north of Hamilton Road and is shown in [Figure 8.](#page-37-0) The general area land use is open range and agricultural fields, with some rural residential surrounding the municipal wastewater treatment site. The site has been permitted by the state since 1989 (Idaho DEQ Land Application Permit LA-000037) to land apply its treated wastewater (DEQ 1996). The waste streams currently collected are from the primarily residential population base; businesses do not contribute any known unusual waste and there are no significant industrial discharges to the treatment system (DEQ 1996). Wastewater treatment consists of a nine cell facultative lagoon system (see [Figure](#page-38-0) [9\)](#page-38-0) that operates in series.

The wastewater treatment system was designed for an average daily flow of 1.71 MGD and a peak hourly flow of 4.9 MGD (DEQ 1996). The lagoons overall occupy 205 acres with a total volume when full of approximately 406 million gallons (DEQ 1996; Keller Associates 2006). The first six clay lined lagoons are treatment lagoons with a 22 day detention time through the system (Keller Associates 2006; DEQ 2009).The last three lagoons store the treated wastewater for up to 214 days during the winter months (Keller Associates 2006; DEQ 2009). Storage lagoons seven (7) and eight (8) are clay lined on the bottom with membrane liner on the sides and storage lagoon nine (9) is fully membrane lined (DEQ 1998; Keller Associates 2006). Storage is followed by disinfection using a chlorine solution directly injected into the pipe through a diffuser; treated effluent

is then sent to be land applied on 260 acres during the growing season of April 1 through October 31 (DEQ 1996; Keller Associates 2006). Irrigation pumps apply effluent by sprinkler in accordance with Irrigation Water Requirements throughout the growing season to fields that historically were planted with small grain, alfalfa, and sugar beets (DEQ 2009; Keller Associates 2006). The permit requires that constituent loading to the site for COD is limited to 50 pounds/acre-day for the growing season and the maximum nitrogen loading is 150% of crop uptake.

In 2005, approximately 500 MG of wastewater entered the treatment system and 286 MG were land applied (Keller Associates 2006). The system was designed for lagoon seepage to occur (Keller Associates 2006) and during this study seepage was estimated to be 200 million gallons per year (Keller Associates 2006; DEQ 2009). Only eight lagoons were in operation during sampling as lagoon six was taken offline in 2002 due to lagoon seepage in excess of the state rule limit of 0.250 inches (6800 gallons) per acre per day (DEQ 1998).

The wastewater treatment site has five monitoring wells (MW) and is shown in [Figure 10.](#page-39-0) The wells were all drilled to depths of less than 20 feet where basalt rock was encountered (Keller Associates 2006; Mountain Home 1991). MW 1, MW 4, and MW 5 have all been dry as they have never encountered ground water (Mountain Home 1991). MW 1 is 20 feet below the ground surface, MW 4 is 12.5 feet below the ground surface, and MW 5 is 10.5 below the ground surface (Mountain Home 1991). MW 2 and MW 3 both have had water present since being drilled, MW 2 is 14 feet deep and MW 3 is 12 feet deep (Mountain Home 1991). The Mountain Home Land Application site

Engineering Reports on file with DEQ and potentiometric contours maps of the regional and perched water table show a south to southwesterly direction of ground water flow [\(Figure 11](#page-40-0) and 12) (Keller Associates 2006; Arney et al. 1984; Bendixsen 1994; Shervais et al. 2002; DEQ 2009; Young et al. 1992). The contours support that MW 1 is upgradient from the lagoons; MW 2 and MW 3 are downgradient from the lagoons and upgradient of MW 4 and MW 5 [\(Figure 10\)](#page-39-0).

Figure 8 Mountain Home South Study Area with Township, Range and Section Overlay Showing City of Mountain Home Wastewater Treatment Lagoons (adapted from Baldwin et al. 2009)

Figure 9 City of Mountain Home Wastewater Treatment Facility Lagoons (adapted from Google Earth 2007)

Figure 10 Monitoring Wells at Mountain Home Wastewater Treatment Facility

Figure 11 Potentiometric Contours on the Regional Water Table, Mountain Home Area (adapted from Young et al. 1992); Black Outline Shows General Location of Mountain Home Wastewater Treatment Facility (adapted from Baldwin et al. 2009)

Figure 12 Potentiometric Contours on the Perched Water Table, Mountain Home Area (adapted from Young et al. 1992); Black Outline Shows General Location of Mountain Home Wastewater Treatment Facility (adapted from Baldwin et al. 2009)

Geology

The City of Mountain Home and the wastewater treatment site are on a southwest sloping broad flat plateau that lies at an elevation of about 3,140 feet above mean sea level on the northeast margin of the western Snake River Plain as shown in [Figure 13](#page-42-0) (Shervais et al. 2002; Arney et al. 1984).

Figure 13 Regional Map of the Western Snake River Plain Showing Mountain Home, the Danskin Mountains to the North and the Mount Bennett Hills to the Northeast (original source Shervais et al. 2002 and adaption taken from Baldwin et al. 2009)

The geological framework of the area provides a context for recharge to the local and regional aquifer system (Baldwin et al. 2009). Rhyolite lavas from volcanic activity form the base layer on which sediments and basalts were deposited (Shervais et al. 2002) [\(Figure 14\)](#page-43-0).

Figure 14 Interpretative Cross Section Across the Western Snake River Plain in the Vicinity of Mountain Home; Line of Section A-A' is Shown on Figure 13 (original source Shervais t al. 2002 and adaption taken from Baldwin et al. 2009)

Shield volcanoes located southwest of Mountain home created a basalt cap over underlying sediments (Shervais et al. 2002; Wood and Clemens 2002). This basalt cap, represented by QTrs on [Figure 15,](#page-44-0) surrounds a topographic low that includes the City of Mountain Home and the wastewater treatment site (Baldwin et al. 2009).

Figure 15 Geologic Map and Explanation of Units for the Mountain Home Area Prepared by Shervais et al. 2002 (original source Shervais et al. 2002 and adaption taken from Baldwin et al. 2009)

Well drillers logs from wells located south of Mountain Home show that the

uppermost basalt is from about 460 to 540 feet thick and is overlain by deposits of clay,

silt, sand and gravel, referred to as Quaternary Alluvium (Qal) on the map (Baldwin et al. 2009). The thickness of the alluvium (ranging up to about 80 feet) reveals the relief of the underlying basalt surface (Baldwin et al. 2009) and was determined from drillers" logs for wells located within the outline of the alluvial deposits as shown in [Figure 16](#page-46-0) (Baldwin et al. 2009).

Figure 16 Map Showing Outline of Qal in the Mountain Home Area and Alluvium Thickness, in Feet, from Selected Wells (original source Shervais et al. 2002 and adaption taken from Baldwin et al. 2009)

This area forms the moat where the perched aquifer exists surrounded by basalt (Shervais et al. 2002; Baldwin et al. 2009). A cross-sectional area of the Qal formation in [Figure 17](#page-47-0) shows alluvial material that lies over the basalt.

Ground water

A shallow perched aquifer and a deep regional aquifer exist in this study area (Norton et al. 1982; Bendixsen 1994; Baldwin et al. 2009). The localized perched aquifer is within the shallow alluvial material mentioned above with the potentiometric contours previously shown in [Figure 12.](#page-41-0) This area underlies about 38,000 acres (Young et al. 1992) that includes the wastewater treatment site area. This aquifer has a depth to ground water of 2 to 40 feet within the shallow sediments and is found mainly in the clay, silt, sand, and gravel of the Quaternary Alluvium (Qal) (Norton et al. 1982). Recharge to the perched aquifer occurs from local creeks, irrigation canals, and a reservoir located north of the study area (Norton et al. 1982; Bendixsen 1994; Baldwin et al. 2009). Seepage from the wastewater treatment lagoons also provides some recharge to the perched aquifer (Norton et al. 1982; Bendixsen 1994). Ground water contour maps from multiple studies of the area between 1968 and 1990 indicate that the direction of ground water movement in the perched aquifer is towards the southwest (Norton et al. 1982; Keller Associates 2006; DEQ 2009; Bendixsen 1994; Young et al. 1992), which includes the

area at the wastewater treatment site as seen in [Figure 12.](#page-41-0) An evaluation of data sets and elevation contours for this perched aquifer from studies from 1968 to 1990 indicates that no significant changes to flow direction or gradient have occurred over this time (Bendixsen 1994). It is assumed that the sources of recharge water and the discharge area remain similar to the conditions from 1968 to 1990. This area according to IDWR was targeted for further hydrological study and due to budget constraints action toward the study is on hold.

Depth to ground water in the deeper regional aquifer is from 200 to 400 feet below land surface. The water is found primarily in basalts and poorly consolidated detrital material (Norton et al. 1982; Bendixsen 1994; Baldwin et al. 2009; Young et al. 1992). The regional aquifer has permeable zones of highly fractured basalt that occur within dense, relatively impermeable flow units. Recharge to the regional aquifer occurs through precipitation in the mountains north of Mountain Home, percolation from ephemeral streams on the plateau, and through percolation from the perched aquifer (Norton et al. 1982; Bendixsen 1994). Ground water flow in the regional aquifer is in a south to southwest direction (Norton et al. 1982; Keller Associates 2006) as shown in [Figure 11.](#page-40-0)

Conceptual Model

In order to evaluate the potential migration of the wastewater into the perched aquifer a conceptual model of the underlying ground water flow regime is necessary. The geological formations in the study area provide a context for discussion of the

hydrogeologic conditions present at this site and are needed to develop a qualitative conceptual model of ground water flow paths. Further, several historic studies of ground water flow within the area are discussed.

Model Boundaries

The hydrogeologic conditions of the site combined with several dry wells around the periphery of the perched aquifer establish boundary conditions where ground water is not present. As a consequence, boundary conditions can be clearly defined to establish ground water flow paths within the perched aquifer on a qualitative basis. A one box model as outlined in [Figure 18](#page-50-0) was chosen, as there is not enough available quantitative information to apply an analytical solution for contaminant transport (such as the Domenico and Schwartz model) (Schnoor 1996). The goal of the conceptual model is to describe in some qualitative manner flow characteristics and associated ground water flow paths in the perched aquifer.

Figure 18 Aerial View of One Box Model of Site System Boundary

The boundaries of the model have been chosen to correspond with known boundary conditions where possible. Water flow in and out of the model boundaries is used to characterize flow within the one box model. A three-dimensional box of the boundaries is shown in [Figure 19.](#page-51-0)

Figure 19 Perspective View of Site System

The boundaries are described below.

Northerly No Flux Boundary

The northerly no flux boundary is a flow line that is dictated by the Potentiometric Contours on the Perched Water Table, Mountain Home Area [\(Figure 12\)](#page-41-0). The northerly no flux boundary is a physical boundary associated with flow paths and extends through Monitoring Well 1 where no ground water is present and follows the south west flow path of the perched aquifer to the extent of the perched aquifer [\(Figure 18](#page-50-0) and 19). This boundary extends vertically from the ground surface approximately 20 feet to the basalt cap. Dry wells, which extend to the basalt layer underlying the perched aquifer, lie along

this boundary. As a consequence, it is assumed that no ground water flows across this boundary.

Southerly No Flux Boundary

The southerly no flux boundary is a flow line that is dictated by the Potentiometric Contours on the Perched Water Table, Mountain Home Area [\(Figure 12\)](#page-41-0). The southerly no flux boundary is a physical boundary associated with flow paths and extends through Monitoring Well 4 [\(Figure 10\)](#page-39-0), where no ground water is present and follows the south west flow path of the perched aquifer to the extent of the perched aquifer [\(Figure 18](#page-50-0) and 19). This boundary extends vertically approximately 10 feet from the ground surface to the basalt cap. As a consequence, no ground water flows in the alluvium of the perched aquifer across this boundary.

Up gradient In-Flux Boundary

The eastern boundary extends horizontally from the eastern edge of the northern boundary to the eastern edge of the southern boundary [\(Figure 19\)](#page-51-0). Based upon the large scale ground water gradients, of approximately 60 ft per mile (Young et al. 1992), ground water flows into the perched aquifer of the conceptual model along this boundary.

Downgradient Boundary

The downgradient boundary [\(Figure 18](#page-50-0) and19) exists to the western edge of the alluvial deposits and thus the western extent of the perched aquifer (Shervais et al. 2002; Baldwin et al. 2009). It extends horizontally from the western edge of the northern no flux boundary to the western edge of the southern no flux boundary. This downgradient boundary extends from the ground surface to the basalt cap. As a consequence of the

alluvium material deposition ending, there is no ground water present as the alluvium of the perched aquifer does not exist along this boundary. It is assumed that no flow exists across this boundary.

Lower Boundary

The lower boundary is defined by the basalt cap underlying the perched aquifer [\(Figure 20\)](#page-54-0). At the western edge of this boundary, the basalt cap rises to the surface and the alluvial sediment ends (Shervais et al. 2002; Baldwin et al. 2009). Based on this information, it is assumed that flows through this boundary, if present, are downward under the influence of gravity since it is not under confining conditions.

Upper Boundary

The upper boundary is defined by the ground surface as shown in [Figure 20.](#page-54-0)

Excluding wells, water crossing this boundary is from evaporation, precipitation, lagoon seepage, and land applied wastewater. As discussed earlier, the 205 acres of lagoons seep approximately 200 million gallons per year into the alluvium of the perched aquifer. This represents a defined inflow into the conceptual model.

Flow Paths

The flow paths across the boundaries of the conceptual model characterize the flow paths for water and PPCPs [\(Figure 20\)](#page-54-0). Water flowing into the system could be from precipitation, the upgradient perched aquifer, and the headworks. Water flowing out with PPCPs could go to the downgradient perched aquifer (through the sludge of the lagoon), be applied on the land application site, and flow from the perched aquifer into the basalt

layer. Based upon the descriptions of the boundary conditions that are defined in the conceptual model, MW 2 exists within the perched aquifer downgradient of seepage from the lagoons. Based upon the descriptions of the boundary conditions that are defined in the conceptual model, MW 3 is not downgradient of the lagoons. The conceptual model and flow paths indicate that MW 2 has the potential to be impacted by PPCPs from the lagoon if PPCPs exist in the perched aquifer. The conceptual model and flow paths indicate that MW 3 exists in the perched aquifer but should not be impacted by any PPCPs originating from the lagoons.

MATERIALS AND METHODS

In this section, the general approach to determine if PPCPs can be contributed to ground water at the City of Mountain Home Municipal Wastewater Lagoon Treatment site will be discussed. This provides a basis for understanding PPCP contamination contributions from wastewater to ground water at the study site.

Local studies on the issue of PPCPs must focus on reconnaissance, which includes collection of baseline data examining PPCP use patterns and related population densities (Focazio et al. 2004). As studies of specific wastewater sites grow, the data may expose relevant trends in PPCP fate and transport over the range of wastewater treatment and hydrologic conditions across the United States. Concentration differences of PPCPs found at this study site in the influent (the headworks) and the effluent (storage lagoon) were evaluated for total removal efficiency. Additionally, concentration differences of PPCPs found in ground water at monitoring wells at this study site were evaluated for total removal efficiency.

Sampling Parameters

The sampling parameters will be discussed, followed by the sampling methods. The target PPCPs for this study are in [Table](#page-24-0) 1 and were chosen based upon analytical capabilities, costs, occurrence data from previous studies (Aga 2008; Clara et al. 2005;

Conn et al. 2006; Drewes 2007; Snyder et al. 2004; Daughten and Ternes 1999; Jos et al. 2003; Daughton 2007), and physio-chemical properties.

The target list includes primarily pharmaceuticals and was initially limited to evaluating carbamazepine, gemfibrozil, sulfamethoxazole, and the estrogens Progesterone, Esterone, Estradiol, and Ethinyl Estradiol 17alpha. Upon evaluation of analytical laboratories capable of testing for these parameters, the list was expanded to include twelve that were part of an analytical panel offered by the chosen lab. The remaining PPCPs were only evaluated for presence or absence in analytical results.

Carbamazepine

Carbamazepine was reviewed to determine its flow through the system and potential as an indicator PPCP. It is unknown if the demographics of the area around the Mountain Home site are such that carbamazepine is used enough to be detectable in the headworks; however, it was chosen due its detection in other studies and low octanolwater partitioning coefficient.

Sulfamethoxazole

Because studies have shown that sulfamethoxazole may be degraded in lakes, it may be correlated to the storage lagoon water at this site. Based upon the limited analytical data and the variance of fate and transport data, it was unknown if sulfamethoxazole could be found in the treatment system or the ground water system in the study area. The prevalence of its use worldwide suggested that it could be found in the headworks if there was enough used in this community.

Gemfibrozil

Since gemfibrozil has a high hydrophobicity and is found frequently in wastewater effluent, it was chosen as a sampling parameter. It is unknown if gemfibrozil will be detectable from this small population in the headworks. If it is present in the headworks, the high hydrophobicity suggests that it may just go directly into the sludge.

Estrogens

Based upon the incomplete removal efficiency of estrogens reported, there is the potential to find estrogens in the headworks and discharge effluent at this site. Four estrogens were tested for in the samples; Progesterone, Esterone, Estradiol, and Ethinyl Estradiol 17alpha (refer to [Table](#page-24-0) 1). Each has a hydrophobicity, which influences a tendency to partition into the hydrophobic organic phase, which suggests presence in the sludge.

Isotopes in Wastewater Treatment Lagoons

The wastewater lagoon treatment is subject to evaporative processes during the time it takes for water to travel through the series of nine lagoons, and as it remains in the ponds for winter storage. Ground water within the site boundary impacted by the lagoons should contain water with a similar isotopic signature as the lagoons, or be representative of water that was mixed with evaporative water from the lagoons (Baldwin and Cook 2004; Baldwin et al. 2009). The oxygen and deuterium isotope results may provide evidence for hydraulic connectivity between surface and ground water based on evaporation (Crosby 1998; Baldwin and Cook 2004; Schnoor 1996; Baldwin et al. 2009). It is expected that the ground water in the regional aquifer would not have been exposed

to evaporative processes and that the headworks for the wastewater treatment system water would have an isotopic signature similar to the regional aquifer since the regional aquifer is the source of water to the city and thus to the wastewater treatment collection system (Baldwin et al. 2009). It is also expected that if water from the lagoons contributes to the perched aquifer, the perched aquifer should have an isotopic signature similar to lagoon water (Baldwin and Cook 2004; Baldwin et al. 2009). A public water system (PWS) well upgradient of the lagoon system was sampled. Water from this well should be similar in isotopic signature to the water in the headworks since evaporative processes are limited as water travels from the well to the wastewater treatment site.

Sampling Methods

Based upon expectation of flow paths [\(Figure 20\)](#page-54-0), four sampling locations were chosen to be sampled in series from the site that is assumed to have the least potential of PPCPs to the site that had the greatest potential for high concentrations of PPCPs. Grab water samples were taken November 2007, following the experimental plan as follows. Samples were taken from Monitoring Well 2, then Monitoring Well 3. Afterward, samples of wastewater were collected from the last wastewater lagoon in series and then near the headworks or influent to the first lagoon.

All personnel on the water sampling team reviewed procedures for using EPA Method 1669 (i.e., "clean hands/dirty hands" procedures) for Sampling Ambient Water for Trace Metals. This protocol was modified for sampling trace PPCPs with appropriate considerations for the ability to contaminant water samples with the PPCPs of interest.

The intent is to maximize the ability of the field sampling team to collect samples reliably and eliminate sample contamination. The collection of samples included a duplicate and field blank for the first four samples taken. The duplicate consisted of sample containers filled with the same composite water from the same sampling site. The duplicates determine both field and laboratory precision. The field blank sample was used to determine the integrity of the field sampling events, the condition of the sample containers supplied by the laboratory, and the accuracy of the laboratory methods. Both the duplicates and blank samples are stored and handled with the normal sample load for shipment to the laboratory. MWH Laboratories [\(Table 2\)](#page-60-0) was used for PPCP sample testing as it met the Unregulated Contaminant Monitoring Regulations (UCMR) Laboratory Approval Program application and Proficiency Testing (PT) criteria. MWH Laboratories is an EPA Approved laboratory for sampling UCMR using EPA 527, 529, 521, 525.2, and 535 methods for PPCP analysis.

The laboratory used for testing of general field parameters is the Idaho Bureau of Laboratories [\(Table 2\)](#page-60-0), which is certified for the chemical and microbiological testing of drinking water from public water systems as part of the primacy agreement between the State of Idaho and EPA. It is certified for microbiological, organic chemistry, and inorganic chemistry analytes and methods.

The laboratory used for isotopes was the University of Arizona Environmental Isotope Laboratory [\(Table 2\)](#page-60-0). This laboratory has been used for isotope testing previously for some of the sample sites and was used for consistency of sample and data evaluation.

At each well, a Geopump Peristaltic Pump was used to draw samples from the wells. Field parameters for specific conductance, pH, temperature, and total dissolved solids were measured with a Horiba Water Quality Checker U-10. The pH and conductivity meters were calibrated to manufacturer specifications, then measurements taken from each well while purging. Field parameter measurements were considered stable when three successive measurements taken at intervals of 5 minutes or more differed by the following: specific conductance 5% or less, pH 0.1 unit or less, and temperature 0.2 ºC or less.

Prior to donning personal protective equipment and obtaining any sample, field personnel designated roles and responsibilities for the sampling and discussed the modified procedures that needed to be used during sampling events. Care was taken not to spill any contaminants (e.g., water, etc.) on or near the sampling site, or to let any sampling equipment come into contact with potential contaminants. Field personnel wore powder-less nitrile laboratory gloves during sampling and processing and changed to

clean gloves with each change in activity or potential glove contamination. They avoided breathing directly over open samples/equipment and avoided direct contact between themselves (including clothing) and the sample, sampling device, and processing equipment. On the day of sampling activities, they avoided contact with or consumption of the products listed below:

- Wastewater compounds such as soaps and detergents, including antibacterial cleansers.
- Pharmaceutical compounds such as prescription drugs, medications, and hormonal substances that are in the list of PPCPs.

Upon arrival at the sampling sites, one member of the two person sampling team was designated as "dirty hands" and the other "clean hands." The persons assumed the "dirty hands" and "clean hands" roles wearing Tyvek coveralls and powder-less nitrile laboratory gloves, and surgical masks with sampling equipment and containers appropriately staged nearby. The dirty hands sampler observed techniques employed by clean hands personnel and to notify the field team immediately in the event that any possible contamination was observed or suspected, or if incorrect sampling techniques were utilized.

The clean hands sampler touched the sample container and transferred the sample from the sample collection device to the sample container. The dirty hands sampler was responsible for the operation of the machinery and other activities that did not involve direct contact with the sample. "Clean hands" placed the clean empty sample containers, removed the lids of the containers, and placed the end of the tubing into the containers.

"Dirty hands" started the pump. "Clean hands" moved the tubing to a clean container. "Dirty hands" stopped the pump. "Clean hands" replaced the lid on the container and returned the container to the designated place. "Dirty hands" placed the bottle into the cooler. Gloves were changed between samples and sample sites. Samples were collected as quickly as is reasonably possible, while carefully preventing any contact between the containers and any surface other than the sampling surface. Lagoon samples were collected by using a grab sampling technique, through immersion into the water by a certified clean amber glass bottle and transferring that into the laboratory prepared bottles. The water in the monitoring well samples was pumped to the surface and the sample containers filled directly. Collected samples were stored in ice chests with freezer packs to maintain 4° C from the time of the sampling event until sample custody was transferred to the lab. After leaving the field, samples going to MWH laboratory were packed for shipment and sealed in ice chests with refrigerated packs, with the required chain of custody forms. Samples going to Idaho Bureau of Laboratories were dropped off by field personnel and samples going to U of Arizona Environmental Isotope Laboratory were shipped.

Some additional isotopes were collected May 2008 from the lagoons for additional verification of isotopic evidence. Laboratories analyzed samples as shown in Table 3. The sample containers were provided by the laboratories in order to ensure that they were clean, uncontaminated, and suitable for the analysis methods used [\(Table 4\)](#page-65-0).

The details are further described in the Quality Assurance Plan (QAPP) that is found in Appendix A and is included within this thesis. The QAPP was based on the EPA

guideline methods available at the time of the original sampling (EPA 2007a; EPA 2007c). The purpose of the QAPP was to ensure that the sample collection and testing was performed appropriately. As fieldwork was conducted, the QAPP document was referenced to maximize the ability of the field sampling team to collect samples reliably and eliminate sample contamination.

Parameters	Test Method	Lab	
Caffeine	USGS 2 mod	MWH Laboratories	
Carbamazepine	USGS 2 mod	MWH Laboratories	
Esterone	USGS 2 mod	MWH Laboratories	
Esteradiol	USGS 2 mod	MWH Laboratories	
Ethinyl Estradiol	USGS 2 mod	MWH Laboratories	
Gemfibrozil	USGS 2 mod	MWH Laboratories	
Ibuprofen	USGS 2 mod	MWH Laboratories	
Iopromide	USGS 2 mod	MWH Laboratories	
Progestrone	USGS 2 mod	MWH Laboratories	
Sulfamethoxazole	USGS 2 mod	MWH Laboratories	
Testosterone	USGS 2 mod	MWH Laboratories	
Trimethoprim	USGS 2 mod	MWH Laboratories	
$\delta 180\%$	IRMS	University of Arizona	
$\delta D\%$	IRMS	University of Arizona	
Total Kjeldahl Nitrogen, TKN	SM 4500-Norg D	Idaho Bureau of Laboratories	
Ammonia as N	SM 4500 NH3 H.	Idaho Bureau of Laboratories	
Nitrogen, Nitrate+Nitrite	EPA 353.2	Idaho Bureau of Laboratories	
Chloride	EPA 300.0	Idaho Bureau of Laboratories	
Sulfate	EPA 300.0	Idaho Bureau of Laboratories	
Total Dissolved Solids	SM 2540C Idaho Bureau of Laboratories		
Total Phosphorus	$10-115-01-1-F$	Idaho Bureau of Laboratories	

Table 3 Laboratory Analysis Method

Sample	Location	Idaho Bureau of Laboratories	U of Arizona Environmental Isotope Laboratory	MWH Labs
Well H_2O	Monitoring Well 2	2 1-liter Polyethylene Cubitainers	1 125 mL Polyethylene Container	Collect 3 Samples: 1 Liter Glass Amber Bottles
Well H_2O	Monitoring Well 3	2 1-liter Polyethylene Cubitainers	1125 mL Polyethylene Container	Collect 3 Samples: 1 Liter Glass Amber Bottles
Effluent H_2O	WW Lagoon 8	2 1-liter Polyethylene Cubitainers	1125 mL Polyethylene Container	Collect 3 Samples: 1 Liter Glass Amber Bottles
Influent H_2O	Headworks/Lagoon 1	2 1-liter Polyethylene Cubitainers	1 125 mL Polyethylene Container	Collect 3 Samples: 1 Liter Glass Amber Bottles

Table 4 Sampling Locations and Containers Used

RESULTS AND DISCUSSION

PPCPs were detected in the samples from the headworks, in the storage lagoon and in a monitoring well downgradient of the lagoon. Twelve PPCPs were tested and eleven were found in the headworks. Seven PPCPs were found in the storage lagoon. Six PPCPs were found in downgradient MW 2. Water isotopes from these samples were examined to further evaluate flow paths.

The sampling results indicate detection of PPCPs in both wastewater and ground water and a summary of the data from the sampling events are found in [Table 5,](#page-67-0) 6, and 7. The raw data can be found in Appendix B. The results indicate that PPCPs are present in detectable and varying concentrations in the samples.

The QAPP defined guidelines for differences within duplicate samples and allowed concentrations in field blanks. These guidelines, when met, provide a measure of accuracy, consistency, and validity of the test results. The results were compared with the QAPP guidelines. Per the QAPP, duplicate samples should be within 30% of each other in order to be valid. All samples, except Caffeine and Esterone, complied with the maximum relative standard difference of 30% as specified in the QAPP. Further discussion of Estrogen related compounds are presented in a section specific to these compounds. Additionally, the QAPP specifies all field blank samples should contain below acceptable values for PPCPs. The field blank samples, except Caffeine, were no-

detect and thus fall within the guidelines specified within the QAPP. As a consequence of the above analysis, all PPCPs test results, except for those of Caffeine and Esterone, are considered accurate.

The PPCP data and isotopic data for all samples will now be discussed to provide a more complete picture of the results. Where connectivity is present between the lagoons and monitoring wells, PPCP concentrations are evaluated for total removal efficiency.

				Sample Location				
		Parameters	Units	PWS Well	Headworks	Lagoon 8	MW ₂	MW ₃
		Temp	$(\text{deg } C)$	18.6			12.5	14.8
		pH	(units)	8.6			6.93	7.24
Field		Sp Cond	(uS/cm)	401			1100	750
	Parameters	D.O.	(mg/L)				0.07	2.67
		turbidity	NTU				$\overline{0}$	$\overline{0}$
Major Ions		Ammonia as N	(mg/L)	0.01	19	8.1	0.03	0.01
		Cl	(mg/L)	16	33.5	67.8	101	31.70
		Nitrogen, Nitrate- Nitrite	(mg/L)	2.5	0.27	0.58	< 0.01	6.00
		Nitrogen, Total Kjeldahl	(mg/L)	0.2	27	14	0.62	0.30
		Total Nitrogen	(mg/L)	2.7	27.27	14.58	0.63	6.3
		Total Phosphorus	(mg/L)	0.02	3.8	$\overline{4}$	0.13	0.27
		Sulfate	(mg/L)	42	22.7	46.9	19.7	111.0

Table 5 Results for Field Parameters and Major Ions from November 2007 Sampling Event

Sample Location PPCP Detect Limit PWS Well Headworks Lagoon 8 $MW2$ **MW2 duplicate MW 3 Field Transfer ng/L Caffeine** 5 ND 7920 48 12 36 2.6 ND **Carbamazepine** 5 ND 92 64 77 82 ND ND **Esterone** 1 | ND | 16 | 450 | 120 | ND | ND | ND **Estradiol** | 1 | ND | ND | 49 | ND | ND | ND | ND **Ethinyl Estradiol 17 alpha** $\begin{bmatrix} 5 \\ 17 \end{bmatrix}$ ND $\begin{bmatrix} ND \\ SD \end{bmatrix}$ ND $\begin{bmatrix} 62 \\ 9 \end{bmatrix}$ ND $\begin{bmatrix} ND \\ ND \end{bmatrix}$ ND **Gemfibrozil** 1 | ND | 440 | 880 | ND | ND | ND | ND **Ibuprofen** 1 ND 1820 ND ND ND ND ND **Iopromide** 5 | ND | 17 | ND | 6.8 | ND | ND | ND **Progestrone** 1 | ND | 520 | ND | ND | ND | ND | ND **Sulfamethoxazole** 1 ND 158 87 1.1 5.8 ND ND **Testosterone** 1 ND ND ND ND ND ND ND **Trimethoprim** 1 | ND | 40 | ND | ND | ND | ND | ND ND: Non Detect

Table 6 Results for PPCPs from November 2007 Sampling Event

Location	Sample Date	$\delta 180\%$	$\delta D\%$	
		(per mil)	(per mil)	
PWS well	$Nov-07$	-15.5	-124	
Headworks	$Nov-07$	-16.0	-124	
Lagoon 8	$Nov-07$	-7.0	-79	
$MW-2$	$Nov-07$	-7.6	-83	
MW ₃	$Nov-07$	-14.5	-116	
Lagoon 7	$May-08$	-1.5	-56	
Lagoon 4	$May-08$	-8.7	-93	
Lagoon 3	$May-08$	-9.9	-99	
Lagoon 2	$May-08$	-11.3	-106	
Lagoon 2	$May-08$	-11.3	-106	
Headworks	$May-08$	-15.9	-125	

Table 7 Isotope Data

Isotopes

Isotope data of oxygen and hydrogen (¹⁸O and ²H) are listed in Table 7 and plotted in [Figure 21.](#page-70-0) The data show an evaporative trend from the headworks through the series of lagoons.

Figure 21 Oxygen Versus Deuterium Isotopes for Samples Collected, Plotted Against the MWL

The headworks and PWS have comparable isotopic values. This confirms that the water from the regional aquifer has undergone little evaporation as it travels from the well head through the distribution system and to the wastewater treatment system. The samples from wastewater treatment lagoons 2, 3, and 4 show a progressive increase in concentration of the heavier isotopes of oxygen and deuterium. This indicates that the water in the lagoons has evaporated as it has passed through the successive lagoons as more ¹⁶O and ¹H has entered the vapor phase. The storage lagoons contain treated water

that is entering for storage, and water that has been accumulating in the lagoon for storage over the winter (or non-irrigation season). The water samples collected from these lagoons show additional evaporative effects and plot in the upper-right part of the graph. The range in oxygen and deuterium values reflects evaporative effects of seasonality and detention time. The storage lagoons are designed so that Lagoon 7 is filled first and then drawn down last, as water is pumped for irrigation in the summer. The isotopic evidence also supports this as shown in [Figure 21.](#page-70-0) The results show that the most evaporation has occurred in Lagoon 7, which has the longer detention time. Lagoon 9 should be the last to fill and the first to empty in the series of three storage lagoons, thus it will have the shortest detention time for all three storage lagoons. The data confirms that lagoon 9 has less evaporative effects than lagoon 7 and 8. MW 2 also shows evaporative effects indicative of surface water from lagoon 9. MW 2 is downgradient of lagoon 9, and isotope results from the lagoon water and the ground water at MW 2 are similar. The isotope evidence indicates that ground water at MW 3 does not contain water that has been exposed to evaporative effects to the same extent as the wastewater treatment and storage lagoons. This is a line of evidence for establishing that the water from MW 3 is not hydraulically connected to water seeping from the pond system. These isotopic data corroborate the ground water flow direction derived from measurement of ground water level in the perched aquifer (shown in [Figure 12\)](#page-41-0) and show that ground water cannot flow from MW 2 to MW 3. The isotopic results from MW 3 indicate that ground water at this well is primarily from sources that are not lagoon surface water. Thus, the ground water flow direction in the perched aquifer is to the west or southwest

from the lagoons and MW 3 is cross gradient to ground water originating from the lagoon system. This isotope data is in agreement with conceptual model.

Carbamazepine

Carbamazepine data [\(Table 8\)](#page-72-0) represented in the column chart shown in Figure 22 indicates that carbamazepine was found in six sampling locations, including MW 2 in the perched aquifer.

Table 8 Carbamazepine Concentrations from Collected Samples

Analyte	PWS	Headworks	Lagoon	MW $\overline{2}$	\vert MW 2 duplicate	MW	Field Transfer
	ng/L	\mathbf{ng}/\mathbf{L}	ng/L	ng/L	ng/L	\mathbf{ng}/\mathbf{L}	ng/L
Carbamazepine	ND	92	64	77	82	ND	ND

Figure 22 Carbamazepine Concentrations from Collected Samples

The average concentration found from all six samples taken was 79.2 ng/L. The carbamazepine concentrations show comparable values (an average of 78 ng/L) through the wastewater treatment process from the headworks to lagoon eight. The total removal efficiency of the wastewater treatment system indicates approximately 30% treatment removal of carbamazepine from the headworks to the storage lagoon. The data from the samples taken from MW 2 indicate an average concentration of 79.5 ng/L. The isotopes and the carbamazepine data indicate that the lagoon water is impacting the local perched aquifer at MW 2. These findings support that carbamazepine shows little attenuation during ground water recharge. The log $[K_{ow}]$ of carbamazepine [\(Table](#page-24-0) 1) is less than 2.5, which, as discussed earlier, would be predictive of having low sorption potential. This data supports the premise that carbamazepine sorption is very low. These findings may also support that carbamazepine can be used as an indicator parameter, to identify and perhaps quantify domestic waste impact to ground water.

There was no detection of carbamazepine in the blank water field transfer and MW 3. The carbamazepine data along with the isotope data further suggests that MW 3 is not downgradient to the lagoon system, and is in agreement with conceptual model.

Sulfamethoxazole

Sulfamethoxazole data [\(Table 9\)](#page-74-0) represented in the column chart shown in [Figure](#page-74-1) [23](#page-74-1) indicates that sulfamethoxazole was found in the headworks, the storage lagoon, and MW 2 in the perched aquifer.

Sulfamethoxazole shows a decreasing concentration from process order in the wastewater treatment lagoons. The trend in concentration from the headworks to the storage lagoon indicates attenuation during treatment, and then further attenuation from

the lagoons to MW 2. Utilizing the supporting evidence that MW 2 is connected to the lagoons based on the information referenced in [Figure 21,](#page-70-0) the sulfamethoxazole data indicates that sulfamethoxazole attenuation occurs from the storage lagoon during transport to MW 2. There was no detection of sulfamethoxazole in the blank water field transfer or in samples from MW 3. The carbamazepine and sulfamethoxazole data along with the isotope data further suggests that MW 3 is not downgradient to the lagoon system and is in agreement with the conceptual model. Sulfa drug removal, as discussed earlier, is shown to be variable and detectable in effluent and this data supports that incomplete removal is occurring. Only about 15-20% of the active drug appears in the urine and this population base of 11,000 provides enough sulfamethoxazole to be detected in the headworks. These data also suggest, approximately 55% total removal efficiency from the headworks to the storage lagoons.

These data show low dose concentrations of the antibiotic sulfamethoxazole in the storage lagoon. As discussed earlier, there is concern over low dose antibiotics in wastewater lagoons and the potential for the spread and maintenance of multi-resistance bacterial pathogens.

Sulfamethoxazole is seeping from the lagoons into the perched aquifer in very small but detectable concentrations that average 3.5 ng/L.

Gemfibrozil

Gemfibrozil data [\(Table 10\)](#page-76-0) represented in the column chart shown in [Figure 24](#page-76-1) indicates that gemfibrozil was found in the headworks and the storage lagoon but not in the perched aquifer.

Analyte	PWS	Headworks	Lagoon	MW ി	MW ₂ duplicate	MW	Field Transfer
	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L	ng/L
Gemfibrozil	ND	440	880	ND	ND	ND	ND

Table 10 Gemfibrozil Concentrations from Collected Samples

Gemfibrozil is found in treated effluent from various wastewater treatment systems and these data show an increasing concentration trend from process order in the wastewater treatment lagoons from the headworks to the storage lagoon. Since

gemfibrozil is excreted as a metabolite, which was not tested for in this study, the increased concentration can plausibly be explained by transformation of metabolites to gemfibrozil in the storage lagoons. Additionally, evaporative effects within the storage lagoons may lead to increased concentration of gemfibrozil. Analysis of total removal efficiency does not apply, as the data shows an apparent increase in concentration during the lagoon treatment process. Gemfibrozil has a high hydrophobicity [\(Table](#page-24-0) 1) with a log $[K_{ow}]$ of 4.77 and its preference to partition to sludge could be explained by this data. There was no detection of gemfibrozil in the blank or monitoring wells.

Estrogens

There were four estrogens analyzed in the sampling and the data [\(Table 11\)](#page-77-0) is shown in the column chart in [Figure 25.](#page-78-0)

Analyte	PWS	Headworks	Lagoon 8	MW	MW ₂ duplicate	MW 3	Field Transfer
	ng/L	\mathbf{ng}/\mathbf{L}	ng/L	ng/L	\mathbf{ng}/\mathbf{L}	ng/L	ng/L
Esterone	ND	16	450	120	ND	ND	ND
Estradiol	ND	ND	49	ND	ND	ND	ND
Ethinyl Estradiol 17 alpha	ND	ND	62	9	ND	ND	ND
Progestrone	ND	520	ND	ND	ND	ND	ND

Table 11 Estrogen Concentrations from Collected Samples

Figure 25 Estrogen Concentrations for Collected Samples

There was no clear overall trend with the estrogens. Esterone, Estradiol, and Ethinyl Estradiol 17alpha show increased concentrations in the storage lagoon. These data are indicative of Esterone, Estradiol, and Ethinyl Estradiol 17alpha undergoing deconjugation of the metabolite during treatment, thus forming the parent compound. There was no analysis for estrogen metabolites, as a consequence a non-detect in the headworks of the parent compound (the estrogen), followed by a subsequent detection in the treatment process, can indicate that metabolites exist. Estimation of a total removal efficiency of this system for estrogens is not possible without the metabolite data. Progesterone was only found in the headworks of the treatment system and was not detected in other samples, which may indicate treatment in that it either breaks down or is partitioning to the sludge during treatment. The log $[K_{ow}]$ of 3.67 [\(Table](#page-24-0) 1) predicts a

medium to high sorption potential and thus partitioning to the sludge during treatment is likely. The sampling data from MW 2 detected only two estrogens, Esterone and Estradiol. There were inconsistencies in concentrations analyzed for estrogens in MW 2. There is no known explanation for this. The sludge or alluvial materials were not studied to determine if estrogens are preferentially partitioning to sludge or breaking down.

Total Nitrogen

The wastewater treatment system is designed for nitrogen removal and the data [\(Table 12\)](#page-79-0) as shown in the column chart in [Figure 26](#page-80-0) indicates that nitrogen removal is occurring from the headworks to the storage lagoon.

Table 12 Total Nitrogen from Collected Samples

Analyte	PWS	Headworks	Lagoon	MW	MW	Field Transfer
	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
Total Nitrogen	2.7	27.27	14.58	0.63	6.3	ND

Figure 26 Total Nitrogen from Samples Collected

Ground water at MW 2 has a very low total nitrogen concentration. A significant decrease in total nitrogen occurs in the vadose zone as seepage moves from the storage lagoon to the perched aquifer. At this site, there is an unknown mechanism by which nitrogen is being removed from the ground water going to this well. By comparison, ground water at MW 3 has a much higher total nitrogen concentration, which indicates that the attenuation is not the same mechanism. This supports previous discussed trends with isotope and PPCP data, which support that MW 3 is not connected to the lagoons, that ground water flow is to the west or southwest from the lagoons, and is in agreement with the conceptual model. The source of nitrogen must originate from areas to the east or northeast.

CONCLUSION

This study confirmed that during the course of wastewater treatment in a lagoon system PPCPs from a small population base are present in inflow to a lagoon treatment system and also within the treatment and storage lagoons. Through development of a conceptual model and isotope sampling data the study provided evidence of hydraulic connectivity between the lagoons and a downgradient ground water monitoring well. Furthermore, some PPCPs were shown to have traveled from the lagoons to a downgradient ground water site, which is in agreement with the conceptual model. The study also suggests that PPCPs may exist as metabolites within the headworks and subsequently reform to the biologically active parent compound at later stages of treatment and in downgradient ground water. This is a critical point when studying compounds such as estrogens, since the metabolites must also be studied in order to form a quantitatively accurate study. The transformations of PPCPs makes studying and quantifying the PPCPs and their metabolites in a treatment system more complicated. The metabolites and their properties may allow for additional environmental pathways of transformation. This study did not include metabolites and, as a consequence, limited the ability of the study to characterize PPCPs, where metabolites may be present. The PPCP detections at this site can help define exposure and add to the data for development of models for predicted environmental concentrations and risk assessment. Where background environmental levels of biologically active PPCPs exist, the impacts from

chronic low dose exposure are relatively unknown in living organisms. Exposure data and toxicity data are important pieces of understanding for developing risk and must include the effects from a specific PPCP and concomitant exposure. Another area for further study is the unintentional exposure of non-target species of PPCPs developed for human health benefit and the environmental cascading effects that may occur from those affected species.

This Idaho study of PPCPs in a municipal wastewater treatment lagoon system could be expanded. Further studies could be directed in two broad categories. The first category could be those studies that concern the specific environment that the PPCPs are traveling within and the second category would be the study of the PPCPs within those travel pathways.

Within the first category, the hydrogeology of the study area could be mapped in greater detail to further refine the model of potentiometric ground water flow. This could be done through the drilling of additional monitoring wells and subsequent monitoring of ground water levels. Additional well data could be used to further characterize the vadose zone and geologic formations at the study site. Another potential area of study would be to examine the plume characteristics within the ground water. This could be accomplished by increasing the number of sampling sites. Studies within this category would also substantiate the assumptions about the subsurface conditions below the lagoons and the ground water flow patterns at this site.

The second category of studies could include a longitudinal study and quantitative analysis of PPCPs and related metabolites in the various stages of treatment along with

dye tracer studies to determine the actual mixing that occurs in each lagoon. The lagoon sludge could be analyzed for PPCPs to further define the assumptions with hydrophobic PPCPs and partitioning to the sludge. The study of this sludge and its life cycle could provide important information about PPCP fate and transport. Sludge is often removed from lagoons and land applied and there is limited research in this area of study to date. The list of PPCPs studied could also be expanded. The regional aquifer downgradient from this site could be explored for impacts from PPCPs and other parameters. The vadose zone below the lagoons could be defined and core samples taken and analyzed for PPCPs.

Additionally, in this second category of study, the liquid and solid streams of exposure could be evaluated based upon their use as nutrients for crops, and assimilation of PPCPs within those crops before and after crop harvest and processing. Human wastewater has nutrients that become more valuable as land resources become limited and agricultural needs grow with the growing world populations. The understanding of risk from the liquid and solid streams generated from wastewater treatment is important to global sustainability as it applies to wastewater reuse. With wastewater reuse, the needs for fresh water sources and ground water recharge must be considered.

As PPCPs primarily enter into wastewater treatment systems through the sewers, reducing the probability of exposure may be an important function of the mitigation efforts that should be explored. Concerned communities can immediately attempt to reduce loading to sewer systems of PPCPs through community drug take back programs.

Studies that begin to identify and quantify the reduced loadings of PPCPs in the sewer would be another avenue for exploration in mitigation.

This study found PPCPs contributed to the environment through municipal wastewater lagoon treatment, which adds to the exposure data and supports that further study of PPCPs is imperative and of concern.

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APPENDIX A

Quality Assurance Project Plan

Monitoring for Pharmaceuticals and Personal Care Products from a Wastewater Treatment Site

Prepared by:

Tressa Nicholas

October 23, 2007

DEQ: WTR.GWP_.ECMH.4004.700

Monitoring: EMERGING CONTAMINANTS - MOUNTAIN HOME

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PROJECT MANAGEMENT

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- Tressa Nicholas, Project Manager, Idaho Department of Environmental Quality, Boise Regional Office
- Ed Hagan, Idaho Department of Environmental Quality, State Administration **Office**
- George Murgel, Professor, Boise State University, Engineering Department
- • Donald Bledsoe, Quality Director Idaho Department of Environmental Quality State Administration Office

Project Description and Objectives

This study involves sampling for a selected set of Pharmaceuticals and Personal Care Products (PPCP's) in wastewater before and after treatment, and from existing wells in the vicinity of a wastewater treatment and land application site. Limited information now exists on the fate of these chemicals in soils and groundwater and this project

provides a contribution specifically for the state of Idaho that investigates groundwater impacts from municipal wastewater sources. Groundwater monitoring from this wastewater treatment and land application site will be a reconnaissance mission to answer the following questions: Are any of the set of PPCPs present? What concentration are they present in? Where were they found?

Purpose of This Study

The purpose of this study is to investigate a municipal wastewater land application site in Idaho to evaluate contributions of Pharmaceuticals and Personal Care Products to groundwater from treated water. The multiple samples from the path of the waste water stream will be analyzed for fifteen PPCP's. These sample data will be used to further characterize the fate of the selected set of PPCP's from municipal waste water effluent. Limited information exists on the fate of these PPCP's in soils and groundwater and this project would provide a contribution specifically for the state of Idaho that investigate impacts of PPCP's from municipal wastewater sources.

Site Criteria

Sites considered were within the state of Idaho. A site that has a history of municipal wastewater application and soils and monitoring well sample results is ideal. A site that has a significant population base, enough to provide a diverse pharmaceutical and personal contaminant load to a treatment system would also be ideal.

Wastewater Treatment Methods

Secondary or tertiary treatment with land application is preferred and lagoons are a common treatment system used prior to land application. There are over 7000 facultative lagoons that serve communities around the United States and a site with facultative lagoons may provide information about fate and transport with this treatment method.

Potential Sites Evaluation

The City of Mountain Home located in Elmore County Idaho may be a good site. The City treats municipal waste water in a series of facultative treatment lagoons and land applies the treated wastewater to approximately 260 acres of farm ground. There are two dedicated monitoring wells down gradient of the treatment lagoons. The site has been permitted for land application since 1989.

The City of Kuna, New Meadows and Calloway Ranch were considered as potential sites. The population base and location of groundwater wells make the City of Mountain Home site a better choice over these three other sites.

Site Identification

The City of Mountain Home sits on a southwest sloping broad flat plateau 3140 feet above mean sea level. The city has a population of about 11,200 people and treats municipal wastewater through lagoon treatment followed by chlorine disinfection and then slow rate land application during the growing season (April 1 through October 31). The site is approximately 2.5 miles south of the city. Eight facultative lagoons with a total volume of 284 million gallons) and an 84 million gallon storage lagoon (s) collect

and treat the wastewater from the city. The wastewater application area is comprised of five management units (MU). Irrigation pumps apply effluent in accordance with crop needs to these fields which are planted with small grain, alfalfa and sugar beets. Chlorination automatically runs when the pumps are in operation. Effluent flows from the storage lagoon and a chlorine solution is injected through an across the pipe diffuser. It then travels about .5 mile to the irrigation pump station. The chlorine contact time is provided in this travel time. The number of irrigation lines in use at any particular time varies based on crop needs and acreage. Disinfected lagoon effluent is applied to a 258 acre land application area.

There are a variety of loam soils on site with varying depths. Depth to ground water in the deep regional aquifer is from 200 to 400 feet below land surface. There is also a localized perched aquifer in the vicinity of the treatment lagoons that has a depth to groundwater of 2 to 40 feet below land surface. This perched aquifer occupies about 38,000 acres and is found mainly in the clay, silt, sand and gravel of the Quaternary Alluvium. The beneficial uses of groundwater in the area are for drinking and agricultural irrigation. Recharge to the regional aquifer occurs through precipitation in the mountains north of Mountain Home, percolation from ephemeral streams on the plateau and through percolation from the perched aquifer. The regional aquifer has permeable zones of highly fractured basalt that occur within dense, relatively impermeable flow units. Groundwater flow in the regional aquifer is generally in a south to southwest direction. Recharge to the perched aquifer occurs from local creeks, irrigation canals and the nearby reservoir. Seepage from the wastewater treatment lagoons also provides some recharge to the

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perched aquifer. The direction of ground water movement in the perched aquifer at the wastewater treatment site is believed to be to the southwest.

Project/Task Description

The purpose of this project is to gain a better understanding of PPCP contaminant contributions from wastewater to groundwater. This monitoring will focus on the water quality sampling of existing ground water wells and nearby sewer lagoons to further characterize impacts by a selected set of PPCPs. Water Samples will be collected over two phases. Phase one will involve taking water samples at four locations to serve as assessments for contributing concentrations of 15 PPCP's. Phase two will be determined after the results have been assessed for phase one. Phase two will involve sampling existing area wells based upon well location, well depth and permission from landowners.

Specific testing and objectives for both phase one and two are as follows:

- Onsite field parameters measurements will be evaluated at all water collection sites to characterize water properties and evaluate purging effectiveness. Field parameters for specific conductance, pH, temperature, and total dissolved solids will be measured.
- Cations and anions will be tested from water samples collected at all monitoring sites to evaluate water chemistry to determine variations of potential source water and contaminants in the area.
- Nitrate testing will be completed for all collections sites to determined extent and boundaries of nitrate contamination.
- PPCP sampling for 15 specific analytes will be conducted to determine potential influence of wastewater streams contribution of PPCP's to the environment.

• Stable isotope testing samples will be collected and archived for each ground water collection site. This testing may provide additional information to delineate nitrate sources, ground water recharge sources, and variations in isotopic chemistry across the area.

Quality Objectives and Criteria for Measurement Data

The objectives of quality assurance and quality control (QA/QC) is to assure that analytical results obtained by sample analyses are representative of actual chemical and physical compositions of the water samples in the field. Project management will be responsible for QA/QC associated with collection of samples, this includes a duplicate and field blank for the first four samples taken in phase one. The duplicate will consist of sample containers filled with the same composite water from the same sampling site. The duplicates will be used to determine both field and laboratory precision. The field blank sample is used to determine the integrity of the field sampling events, the condition of the sample containers supplied by the laboratory and the accuracy of the laboratory methods. Both the duplicates and blank samples are stored and handled with the normal sample load for shipment to the laboratory. Laboratory duplicate samples should be within 30% of each other. Blank samples should contain below their acceptable values of PPCPs.

Phase two details for this project are yet to be determined, and the data from phase one will be used for decisions on further sampling.

Project management will provide assurance that the selected analytical laboratories will use appropriate QA/QC methods. A laboratory that has met the Unregulated Contaminant Monitoring Regulations (UCMR) Laboratory Approval

Program application and Proficiency Testing (PT) criteria will be used for assessment of PPCP's. The laboratory will be approved for UCMR 2 and can only use EPA Approved laboratory for sampling UCMR using EPA 527, 529, 521, 525.2 and 535 methods for PPCP analysis. MWH Laboratories will be used for PPCP sample testing as it has met the above requirements.

The laboratory used for testing of general field parameters is the Idaho Bureau of Laboratories which is certified for the chemical and microbiological testing of drinking water from public water systems as part of the primacy agreement between the State of Idaho and EPA. It is certified for microbiological, organic chemistry and inorganic chemistry analytes and methods.

The laboratory used for isotopes will be the U of Arizona Environmental Isotope Laboratory. This laboratory has been used for isotope testing previously for some of the sample sites and will be used for consistency.

Special Training Needs/Certification

All personnel on the water sampling team will review procedures for using EPA Method 1669 (i.e., "clean hands/dirty hands" procedures) for Sampling Ambient Water for Trace Metals. This protocol will be modified for sampling trace PPCP's with appropriate considerations for the ability to contaminant water samples with the PPCP's of interest. The intent is to maximize the ability of the field sampling team to collect samples reliably and eliminate sample contamination.

Documents and Records

Documentation for this project will include historical data kept on file at DEQ's state office, as well as all documents created for the sampling events addressed under this QAPP. The generated documentation will consist of at least those documents developed for sample collection procedures, a report summarizing the sampling events and results, and this QAPP. Field notebooks and field data sheets will also be kept and included in the project file. This information will be available and reviewed by the project management for quality control purposes. The data will be recorded using these procedures:

- Project data must be recorded directly, promptly, and legibly.
- Field logbook entries must be made in black or blue permanent ink and must be initialed and dated by the person making the entry.
- Changes or corrections to data must be indicated with a single line through the original entry.

A dedicated field logbook will be used to document the following information during each sampling event:

- Purpose of sample event;
- Make and model of all equipment used;
- Name of sampling personnel and/or field crew;
- Identification of the sampling site (e.g., GPS coordinates, benchmark location, etc.);
- Weather conditions and time of arrival at site;
- Brief description of site conditions, and sampling setup;
- Date and time of sample collection;

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- Sample identification numbers and parameters requested;
- Field parameter measurements and methods;
- All relevant observations pertaining to each sampling event.

DATA GENERATION AND ACQUISITION

Sampling Process Design (Experimental Design)

Site and Sampling Procedure

The wastewater treatment area for the City of Mountain home was chosen for Phase One. Four sampling locations were chosen to be sampled in series from the site that is assumed to have the least potential of PPCPs to the site that may have the greatest potential for highest concentration of PPCPs. Samples will also be taken from Monitoring Well 2 then Monitoring Well 3. Afterward, grab samples of wastewater will be collected from the last wastewater lagoon in series and then near the head-works or influent to the first lagoon. Based on the data collected from phase one of sampling, additional locations for sampling will be proposed for phase two, and future revisions to this QAPP.

At each location, field parameters for specific conductance, pH, temperature, and total dissolved solids will be measured. The pH and conductivity meters will be calibrated to manufacturer specifications, then measurements will be taken from each well while purging. Field parameter measurements will be considered stable when three successive measurements taken at intervals of 5 minutes or more differ by the following:

specific conductance 5% or less, pH 0.1 unit or less and temperature 0.2 °C or less, after which three sets of samples will be taken to be sent to three different laboratories for each sampling site.

TABLE 1. LABORATORIES

TABLE 2. SAMPLING CONTAINERS

Sampling Methods

The sample procedures described below are designed to very conservatively minimize the potential for contamination of the samples. The procedures may be changed based on field experiences and future discussions, and minor deviations from these procedures will not necessarily invalidate the samples but should be noted within the field logbook.

All field personnel must be extremely conscientious with respect to field and sampling techniques while involved with or around collection of samples. Constant care and consideration must be exercised to avoid any contamination of the sampling sites or sample containers. Prior to donning personal protective equipment (PPE) and/or obtaining any sample, field personnel should designate various roles and responsibilities

for each member of the sampling team and discuss procedures to be utilized during sampling events. Care should be taken not to spill any contaminants (e.g., water, etc.) on or near the sampling site, or to let any sampling equipment come into contact with the potential contaminants.

Field personnel will wear powder-less nitrile laboratory gloves during sampling and processing. They will change to clean gloves with each change in activity or potential glove contamination. They will avoid breathing directly over open samples/equipment. They will avoid direct contact between themselves (including clothing) and the sample, sampling device, and processing equipment. Field personnel have been briefed that clothing is a source of detergents, fragrances, and fire retardants. On the day of sampling activities, they will avoid contact with or consumption of the products listed below:

- Wastewater compounds such as soaps and detergents, including antibacterial cleansers. The laboratory analysis of wastewater includes triclosan, an active ingredient in most antibacterial soaps. Triclosan is also commonly found in some deodorants, toothpastes, mouthwashes, skin creams, lotions, laundry detergents, and dish soaps.
- Pharmaceutical compounds such as prescription drugs, medications, and hormonal substances that are in the list of PPCP's.

Upon arrival at the sampling sites one member of the two person sampling team is designated as "dirty hands" and the other "clean hands." The personnel assuming the "dirty hands" and "clean hands" roles will don Tyvek coveralls and powderless nitrile laboratory gloves, and surgical masks with sampling equipment and containers appropriately staged nearby. The dirty hands sampler will observe techniques employed

by clean hands personnel and should notify the field team immediately in the event that any possible contamination is observed or suspected, or if incorrect sampling techniques are utilized.

The clean hands sampler will touch the sample container and transfer the sample from the sample collection device to the sample container. The dirty hands sampler will be responsible for the operation of the machinery and all other activities that do not involve direct contact with the sample. "Clean hands" places the clean empty sample containers, removes the lids of the containers and places the end of the tubing into the containers. "Dirty hands" starts the pump. "Clean hands" moves the tubing to a clean container. "Dirty hands" stops the pump. "Clean hands" replaces the lid on the container and returns the container to the designated place. "Dirty hands" places the bottle into the cooler. Gloves are changed between samples and sample sites. After each sample is collected, the sample number is documented in the field log along with observations by "Dirty hands".

Samples should be collected as quickly as is reasonably possible, while carefully preventing any contact between the containers and any surface other than the sampling surface. Lagoon samples are collected by using a grab sampling technique, through immersion into the water by a certified clean amber glass bottle and transferring that into the laboratory prepared bottles. The monitoring well samples will be collected using a peristaltic pump. The water will be pumped to the surface and the sample containers filled directly. Care will be taken to avoid breathing over the sample containers and masks will be worn. If any contamination of the sample container occurs or is suspected prior to, during, or after sampling, the container in question should not be used in the

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sampling event, and will be replaced with a new, uncontaminated sample container. Care will be taken to avoid contaminating the equipment on site.

Collected samples will be stored in ice chests with freezer packs to maintain $4^{\circ}C$ from the time of the sampling event until sample custody is transferred to the lab. MWH labs will provide ice chests designated for their samples. DEQ ice chests with ice will be used for samples designated for Idaho Bureau of Laboratories and U of Arizona Environmental Isotope Laboratory. After leaving the field, samples going to MWH laboratory will be packed for shipment and sealed in ice chests with refrigerated packs, with the required chain of custody forms. Samples going to Idaho Bureau of Laboratories will be dropped off by field personnel and samples going to U of Arizona Environmental Isotope Laboratory will be shipped.

Sample Handling and Custody

Samples will be directly transported to DEQ headquarters in Boise in coolers with cold packs that were previously frozen. These coolers will be shipped overnight to MWH Laboratories. All proper chain of custody forms will accompany samples throughout the duration of the shipping process.

After the samples have been collected, they will be placed in an ice chest on freezer packs, and held under chain of custody until they are hand delivered to the shipping agent. A chain of custody form will accompany each shipment during transport to the lab. Each ice chest will be sealed just prior to release of custody, with a completed

and properly signed chain of custody form affixed to the inside of the ice chest. The chain of custody form will include the following information:

- Project name/code;
- Client's name and address;
- Laboratory name and address;
- Sample identification number;
- Date and time of collection;
- Type of sample, number of containers, and analysis requested;
- Sample preservation methods;
- Field information and remarks;
- Sample location;
- Calibration information and procedures;
- Signature of sample collector(s);
- Inclusive dates of possession.

The following procedures will be followed to ensure proper chain of custody:

- The sample and seal information checked to verify that they match the chain of custody form;
- The chain of custody record will be checked for a signature;
- A laboratory sample number will be assigned;
- The sample will be stored in a secure area until it is analyzed.

Analytical Methods

Three different laboratories will be used to analyze the samples. See the following tables:

TABLE 3. MWH LABORATORIES PPCP PARAMETERS AND ACCURACY

TABLE 4. U OF ARIZONA ENVIRONMENTAL ISOTOPE LABORATORY

TABLE 5. IDAHO BUREAU OF LABORATORIES

Quality Control

Quality control checks include internal checks for sampling analysis activities, duplicate samples and field blanks. Quality Control procedures for the laboratories have been certified through the US EPA. The following list documents key components of the QA/QC program for this project.

• Laboratory: The PPCP analytical laboratory will provide appropriate samples containers, chain of custody forms, sample labels, and any necessary container seals. A laboratory QA/QC report with continuing calibration checks will accompany each data report and will be stored in the DEQ state office. DEQ will

provide the sample containers that are approved for use in the other two laboratories.

- Sample Collection: All QA/QC procedures for sample collection will be followed by sampling personnel. For this sampling project, the QA/QC procedures will be fulfilled by adhering to all requirements of Section 2 and such adherence will be demonstrated through appropriate documentation of sampling procedures within the field logbook. A standard field logbook, dedicated solely for use in this project, will be kept for all sampling events and will follow the format described in Section 2.
- Field Duplicates: A duplicate sample will be collected and prepared at one of the four sampling sites. All relevant information will be recorded for the duplicates in the field logbook. The duplicates will allow assessment of repeatability and accuracy of the sampling procedures and will also be used to assist in assessment of any cross-contamination issues. Results from the field duplicate analysis will be presented in the analytical report.
- Field Blank: The purpose of the field blank is to assess the amount, if any, of the analytes as contaminants that could be collected in sample containers while open and exposed during sample collection procedures. The field blank will be prepared with a standard supplied by MWH laboratories and shipped from the lab with other sample bottles. The field blank will be sent for analysis with other samples collected for analysis, and results of the field blank analysis will be included in the analytical report.

Personnel will conduct a field blank procedure at one site, during collection of a sample. The same procedures and clean hands/dirty hands techniques used for sample collection will generally be observed in handling and preparing the field blank. It is critical that the field blank container be isolated from contact with any surfaces or other

possible sources of contamination that would have been isolated from contact or exposure to the sample containers.

The clean hands sampler will carefully accept an empty sample container, designated as the field blank container, from the dirty hands sampler, and will then open and place the container in an appropriate location determined by field personnel. The actual location to be used will be determined in the field, based on actual site parameters and conditions. The field blank container will be maintained in the vicinity of the composite sample collection location, but must be isolated from any situation or location that might reasonably be expected to skew or invalidate the purpose of the field blank. The clean hands sampler will fill the field blank container with 1 L of standard water supplied by MWH Labs prior to collection of the well sample for analysis. The well sample procedure will then be conducted per required techniques, exercising care to avoid any unnecessary actions that may cause interference with the field blank assessment. The field blank container will be kept open throughout the sample collection procedure. After the sample has been collected and the container closed, the clean hands sampler will seal and then relinquish the field blank container to the dirty hands sampler. The identification number of field blank container shall be recorded in the field logbook, along with all other required sample information.

Instrument/Equipment Testing, Inspection, and Maintenance

Procedures and schedules for preventive maintenance of sampling equipment are the responsibility of project management. Each instrument or item of laboratory

equipment will be maintained periodically to ensure accuracy. These procedures and frequency of performance are designated in the individual instrument manuals.

TABLE 6. EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Instrument/Equipment Calibration and Frequency

Each instrument or piece of equipment will be calibrated and maintained to ensure accuracy within specified limits. The calibration of analysis instrumentation used under this project will be the responsibility of the technical personnel assigned to the project. The equipment used to collect samples will be calibrated according to manufacturers' procedures or internal guidelines at recommended intervals. Calibration sheets that contain the calibration procedures and the results of each calibration, or the equivalent, will be kept on file. The calibration sheets will also serve as a permanent record of maintenance for the sampling equipment.

TABLE 7. EQUIPMENT CALIBRATION REQUIREMENTS

Inspection/Acceptance of Supplies and Consumables

Sample containers, labels and associated preservatives will be provided by MWH laboratories. Instructions provided by MWH laboratories will be followed throughout the duration of the project.

Data Management

The sampling team is responsible for collection, storage, and transport of field data to the office. A standard system for sample labeling and correlation with appropriate field notes and QC checks will be developed. Laboratory and field data will be compiled by project management.

Hard copies of all field notes and field data sheets will be kept on record at the DEQ office. All information pertaining to this project will be stored in the source files, and electronic copies of all reports will be available for review upon request.

ASSESSMENT AND OVERSIGHT

Assessments and Response Actions

The purpose of assessment is to ensure that this plan is implemented as prescribed.

The quality director will have the lead role in assessing the QA and QC measures employed in this study (e.g. review of sampling procedures). The two project mangers will work together to determine appropriate QA and QC measures. They will also have the lead role in data quality review. Both will work together to assure overall project objectives are met.

Reports to Management

The final field report will be prepared by the project manager and will include:

- A summary of the field work conducted
- The results of the laboratory analyses, including quality assurance measures
- A QA and QC summary
- Conclusions

No specific action will be required by any recipient of the report.

DATA VALIDATION AND USABILITY

Data Review, Verification, and Validation

The objectives of this project are to provide information to determine if any of the set of PPCPs present is present in the wastewater and groundwater at this site? What concentration are they present in? Where were they found? This important information for validation concerns the sampling methods. Verification requirements concern the quality of the actual data and interpretation of the data. Compliance with sampling protocol, record keeping, labeling as defined in this document constitute the requirements for verification.

Verification and Validation Methods

The project managers will be responsible for reviewing the project, collecting samples, labeling, storage, transport, sending the samples for analysis and summarizing the data. They will confirm that the data generated complied with the specifications fo the procedures and objectives of the proposal. Any deviations will be noted and whether to reject, accept or qualify the data will be determined. Data will be subject to visual

inspection and any questions as to values or sample identity will be resolved via line-byline confirmation with the analyzing laboratory.

Reconciliation with User Requirements

Data and conclusions will be peer reviewed at both at DEQ and Boise State

University. The peer-review process is set up to identify technical and scientific concerns.

REFERENCES

Department of Environment Quality DEQ Publication, "An Evaluation of Bacteria in Ground Water Near Mountain Home, Elmore County, Idaho", March 1996

2006 Northwest Regional Wastewater Reuse Conference Papers

Environmental Protection Agency EPA

United States Geological Survey USGS

World Health Organization WHO

National Institute of Environmental Health Sciences NIEHS

APPENDIX B

Project Data

Monitoring for Pharmaceuticals and Personal Care Products from a Wastewater Treatment Site

MWH Laboratories 750 Royal Oaks Drive, Monrovia, CA 91016 PHONE: 626-386-1100/FAX: 626-386-1101

ACKNOWLEDGMENT OF SAMPLES RECEIVED

Idaho Dept of Environmental Quality Customer Code: IDAHODEQ Boise Regional Office Group#: 222609 1445 N. Orchard Boise, ID 83706 Project#: EDC Attn: Tressa Nicholas Proj Mgr: Allen Glover Phone: 208-373-0116 Phone: 916-374-8030

The following samples were received from you on 11/15/07. They have been scheduled for the tests listed beside each sample. If this information is incorrect, please contact your service representative. Thank you for using MWH Laboratories.

 $\mathcal{C}^{\mathbf{R}}$

750 Royal Caks Drive, Suite 100
Microcyte, Californa 91018-0029
Tel. 626 396 1100
Fex: 626 396 1101
1 800 506 LABS (1 601 586 5257)

Laboratory Report

for

Idaho Dept of Environmental Quality Boise Regional Office

1445 N. Orchard

Boise, ID 83706

Attention: Tressa Nicholas Fax: 208-373-0287

MAG Matthew Allen Glover Project Manager

Report#: 222609 Project: EDC

Laboratory certifies that the test results meet all NELAC requirements unless noted in the Comments section or the Case Narrative. Pollowing the cover page are QC Report, QC Summary, Data Report, Hits Report, totaling 7 page [s].

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ACKNOWLEDGMENT OF SAMPLES RECEIVED

The following samples were received from you on 11/15/07. They have been scheduled for the tests listed beside each sample. If this information is incorrect, please contact your service representative. Thank you for using MWH Laboratories.

Laboratory Hits Report #222609

750 Poyal Casa Drive, Suite 100
Morrovil, California 91018-3629
Tel: 626 386 1100
Fax 626 366 1.485 (1 800 566 5227)

Idaho Dept of Environmental Quality Tressa Nicholas Boise Regional Office 1445 N. Orchard Boise, ID 83706

Samples Received Samples 11:56:17

SUMMARY OF POSITIVE DATA ONLY.

Hits Report - Page 1 of 1

Laboratory Data Report #222609

Samples Received

 $11/15/07$

750 Royal Calvis Drive Suite 100
Manuwat, California (#1016-3629)
Tel 626 386 1100 Fax: 420 506 110 1 600 566 LARS (1 800 566 5227)

Idaho Dept of Environmental Quality Tressa Nicholas Boise Regional Office 1445 N. Orchard Boise, ID 83706

Prepared Analyzed OC Refs Method Amalyte **Beault Units** went. Dilution Sampled on 11/14/07 12:50 4 (LAGOON 8) (2711150353) EDC screen by LC-MS-MS I LC-MS-MS) Acetaminophee SD. $nq/1$ 1.4 $\mathbbm{1}$ 11/30/07 11/37/07 14:24 400001 $49/1$ $1 - 4$ 11/20/07 11/27/07 14:24 400001 1 14"-MG-MD 1 Caffeine 48 \mathbf{L} 0.6072 5.07 11/30/07 11/27/07 14:24 400001 (LC MS-MS) Carbanacepins Ł4 $\mathbbm{1}$ $mg/1$ ti ti 11/20/07 11/27/07 14:24 400001 1 LC-ME-ME) Esterone 450 \mathbf{r} $m₀ r₁$ 1.8^{+} 11/20/07 11/27/07 14:24 400001 : Estradiol 1 LC-355-MD 45. \mathbb{Z}^2 \sim 11/20/07 11/27/07 14:24 400001 $1 - 145 - 195 - 145$) Ethinyl Estradiol -17 alpha 62 $ma/1$ \mathbb{R}^3 1.8 11/20/07 11/27/07 14:24 400001 $1 - 2C - 165 - 165$ 1 Pluggetine MD. $na/1$ \mathbf{r} 1 Gentinessit 445 $ma/1$ bd. 16 11/20/07 11/27/07 14:34 400001 1 LC-R0-NS 11/20/67 11/27/07 14:24 400001 | LC-MS-MS 1 Ibuprufen $_{85}$ $mp/1$ $1 - 4$ ı. 11/20/07 11/27/07 14:24 400001 (LC-MB-MB $3D$ 5.0) inpromide $ma/1$ $\overline{1}$ 11/20/07 11/27/07 14:24 400001 1.8 1 LC-HG-HG 1 Progesterons sm $mg/1$ \mathbb{R} 1 60-88-88 11/20/07 11/37/07 14:24 400001) Bulfamethonazole 87 $na/1$ 1.8 y. wo $ng/1$ 10 11/20/07 11/37/07 14:24 400001 $1.10 - 0.01 - 0.01$ 1 Testosterone \leq **HD** 5.0 11/30/07 11/37/07 14:24 400001 (LC-MS-MS ! Triclosan $na/1$ 'n. ab. $f\$ gen 1.0 11/20/07 11/37/07 14:24 400001 | LC-MB-ME : Trimethoprie x sta. 4 Red L L ^C - MB - PB) Caffeine-C1J (70-130) Sampled on 11/14/07 13:40

5(HW) (2711150354)

EDC screen by LC-MS-MS

Laboratory Data Report #222609

T85 Floyd Cale Cone, Suite 105
Marcore, California 91819-0039
Tu: 405 366 1110
Fax 105 365 1110
1 600 569 1405 1110

Idaho Dept of Environmental Quality (continued)

6(MW2-FT) (2711150355) Sampled on 11/14/07 12:30

EDC screen by LC-MS-MS

Data Report - Page 2 of 2

Laboratory QC Summary #222609

750 Royal Caks Orive, Suite 100
Monrevia, California 91018-3629
Tal: 626 366 1100
Fax: 626 366 1101
1 800 566 LABS (1 600 566 5277)

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Idaho Dept of Environmental Quality

QC Summary - Page 1 of 1

Laboratory QC Report #222609

150 Royal Cake Drive, Solid 100
Martinia, California 91016-2029
Tel: 628 200 1100
Fax 626 1455 (1 doll 546 5277)

Idaho Dept of Environmental Quality

OC Ref #400001 EDC screen by LC-MS-MS

Spiess which asceed Limits and Hathod Blanks with positive results are highlighted by Undarlining, Criteria for MS and DUP are edvisory only. batch control is based on LCS. Criteria for duplicates are advisory only. unless otherwise specified in the method.

QC Report - Page 1 of 3

750 Playel Oaks Drive, Solite 100
Marxovie, California 91016-3629
Tel: 828 388 1100
Fax 528 366 1101
1 800 556 LABS (1 800 566 5227)

Idaho Dept of Environmental Quality (continued)

Spikes which exceed Limits and Method Blanks with positive results are highlighted by Onderlining Criteria for HE and DUP are edvisory only, hatch contiol is based on LCS friteria for duplicates are advisory only, unless otherwise specilied in the mainod.

Laboratory QC Report #222609

750 Playst Oaks Drive, Suite 100
Mervove, California 81016-0229
Ter 405 306 1100
Fax: 405 366 1491
1 800 566 1.491 (1 800 546 5227)

Idaho Dept of Environmental Quality (continued)

Spikes which exceed Limits and Method Blanks with positive results are highlighted by Underlining. Criteria for MS and DUP are advisory only, batch control is based on LCE. Criteria for duplicates are advisory only, unless otherwise specified in the method.

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MWH Laboratories 750 Royal Oaks Drive, Monrovia, CA 91016 PHONE: 626-386-1100/FAX: 626-386-1101

ACKNOWLEDGMENT OF SAMPLES RECEIVED

Idaho Dept of Environmental Quality Customer Code: IDAHODEQ Boise Regional Office Group#: 222619 1445 N. Orchard Boise, ID 83706 Project#: EDC Attn: Tressa Nicholas Proj Mgr: Allen Glover Phone: 208-373-0116 Phone: 916-374-8030

The following samples were received from you on 11/15/07. They have been scheduled for the tests listed beside each sample. If this information is incorrect, please contact your service representative. Thank you for using MWH Laboratories.

750 Royal Dalis Drive, Suite 100
Monrovie, California 91018-0825
Fax: 628 386 1101
Fax: 628 386 1101
1 600 560 LABS (1 800 566 5227)

Laboratory Report

for

Idaho Dept of Environmental Quality Boise Regional Office

1445 N. Orchard

Boise, ID 83706

Attention: Tressa Nicholas Fax: 208-373-0287

MAG Matthew Allen Glover Project Manager

Report#: 222619 Project: EDC

Laboratory certifies that the test results meet all NELAC requirements unless noted in the Comments section or the Case Narrative. Following the cover page are QC Report, QC Summary, Data Report, Hits Report, totaling 7 page [s].

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MWH Laboratories 750 Royal Oaks Drive, Monrovia, CA 91016 PHONE: 626-386-1100/FAX: 626-386-1101

ACKNOWLEDGMENT OF SAMPLES RECEIVED

Idaho Dept of Environmental Quality Boise Regional Office Customer Code: IDAHODEQ 1445 N. Orchard
Boise, ID 83706 Group#: 222619 Project#: EDC Attn: Tressa Nicholas Proj Mgr: Allen Glover Phone: 208-373-0116 Phone: 916-374-8030

The following samples were received from you on 11/15/07. They have been scheduled for the tests listed beside each sample. If this information is incorrect, please contact your service representative. Thank you for using MWH Laboratories.

Laboratory **Hits Report** #222619

750 Royal Case Orive, Suite 100
Marsovia, Californe 91010-3629
Fax: 626 366 1102
Fax: 626 366 1102
1 600 566 1.455 (1 600 566 5227)

Idaho Dept of Environmental Quality Tressa Nicholas Boise Regional Office 1445 N. Orchard Boise, ID 83706

Samples Received 15-nov-2007 15:39:26

SUMMARY OF POSITIVE DATA ONLY.

Hits Report - Page 1 of 1

Laboratory Data Report #222619

Samples Received

11/15/07

750 Royal Cake Dine, Switz 105
Mercure, Californe 31015-3229
Tai, 405 366 1100
Fax: 626 386 1101
1 830 586 1.455 (1 800 566 5227)

Idaho Dept of Environmental Quality Tressa Nicholas Boise Regional Office 1445 N. Orchard Boise, ID 83706

! :buproten

11/20/97 11/27/07 14:10 409001 / LC-MS-MS / Flucketine

11/20/07 11/77/07 14.18 400001 / LC HS HS / GenEibrox11

11/20/07 11/27/07 14:18 400001 / LC MS MS

Data Report - Page 1 of 2

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NO

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mep/ I

zna/12
Laboratory Data Report #222619

750 Royal Oaks Orive, Suite 100
Mantovia, California 91016-3829
Tel: 628 366 1100 Fex: 628 385 1101
| 500 586 1.485 (1 800 586 5227)

Idaho Dept of Environmental Quality (continued)

3 (MW 2) (2711150404)

Sampled on 11/14/07 12:50

EDC screen by LC-MS-MS

Laboratory QC Summary #222619

750 Royal Owis Dires, Suite 100
Montova, California 91016-3639
Tel: 626 386 1100
Fax. 659 386 1101
1 600 586 LASS († 600 566 5227)

Idaho Dept of Environmental Quality

QC Summary - Page 1 of 1

Laboratory OC Report #222619

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750 Royal Cake Ome, Suite 100
Movrovis, California 91016-3229
Tal. 626 386 1100
Fax: 626 386 1101
1 800 566 LABS (1 800 566 5227)

Idaho Dept of Environmental Quality

OC Ref #400001 EDC screen by LC-MS-MS

Spikes which exceed Limits and Method Blanks with positive results are highlighted by Underlining. Criteria for MS and DUP are advisory only, batch control is based on LCS. Criteria for duplicates are advisory only, unless otherwise specified in the method.

750 Royal Oaks Drive, Suite 100
Movrovia, California. 91015-3625
Ter. 628 366 1100
Fax. 628 366 1485 (1 600 566 5227)

Idaho Dept of Environmental Quality (continued)

spikes which exceed Limits and Method Blanke with positive results are nighlighted by Underlining, Criteria for MS and DUP are advisory only, basch pubtrol is based an LCS Criteria for duplicates are advisory only, unless otherwise specified in the mathod.

Laboratory QC Report #222619

750 Royal Oals Orive, Suite 100
Vermova, California : 91016-3629
Tel: 605 366 1100
Fax: 626 366 1101
1 800 566 LABS († 800 566 5227)

Idaho Dept of Environmental Quality (continued)

Spikes which exceed Limits and Method Blanks with positive results are highlighted by Underlining. Criteria for MS and DUP are advisory only, batch control is based on LCS. Criteria for duplicates are advisory only, unless otherwise specified in the method.

2220 Old Penitentiary Road Boise, Idaho 83712 Phone: (208)334-2235 Fax: (208)334-2382

Richard F. Hudson, PhD, Chief

 $t_{\rm in}$, $\lambda\tau_{\rm in}$

EPA Lab ID00018 **CLIA Certified Lab**

October 25, 2008

Report To Joe Baldwin Department of Environmental Quality - State Office 1410 N Hilton St Boise, ID 83706-1255

Sample Submitted by Department of Environmental Quality - Boise Regional Office

RE: Workorder E080900074 090908

Profile 8403 - Mt Home - Emerging Contaminants

Enclosed are the analytical results for sample(s) received by the laboratory on Tuesday, September 09, 2008. Results reported herein conform to the most current EPA standards, where applicable, unless otherwise narrated in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

beauger Bac

Wally L. Baker

3CE)

OCT 3 0 2008 **DEPARTMENT OF ENVIRONALEMTALQUALITY BOIS NALOFFICE**

Enclosures

Report ID: 24517 - 303971

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CERTIFICATE OF ANALYSIS

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Richard F. Hudson, PhD, Chief

EPA Lab ID00018 **CLIA Certified Lab**

Workorder: E080900074 090908

SAMPLE SUMMARY

CASE NARRATIVE

2220 Old Penitentiary Road Boise, Idaho 83712 Phone: (208)334-2235 Fax: (208)334-2382

EPA Lab ID00018 **CLIA Certified Lab**

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2220 Old Penitentiary Road Boise, Idaho 83712 Phone: (208)334-2235 Fax: (208)334-2382

Richard F. Hudson, PhD, Chief

EPA Lab ID00018 **CLIA Certified Lab**

CASE NARRATIVE

Lab ID: E080900074-012

Sample ID: 42

SAMPLE Sample Type:

Sample was ran for total dissolved solids past expiration date, results may be affected.

Report ID: 24517 - 303971

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EPA Lab ID00018 **CLIA Certified Lab**

E080900074 090908 Workorder:

ANALYTICAL RESULTS

Report ID: 24517 - 303971

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EPA Lab ID00018 **CLIA Certified Lab**

Workorder: E080900074 090908

ANALYTICAL RESULTS

Report ID: 24517 - 303971

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EPA Lab ID00018 CLIA Certified Lab

Workorder: E080900074 090908

ANALYTICAL RESULTS

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EPA Lab ID00018 **CLIA Certified Lab**

Workorder: E080900074 090908

ANALYTICAL RESULTS

Chemistry samples are disposed of after 45 days unless the laboratory is notified otherwise.

Report ID: 24517 - 303971

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EPA Lab ID00018 CLIA Certified Lab

Workorder: E080900074 090908

ANALYTICAL RESULTS

Report ID: 24517 - 303971

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EPA Lab ID00018 **CLIA Certified Lab**

Workorder: E080900074 090908

ANALYTICAL RESULTS

Report ID: 24517 - 303971

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Page 10 of 16

2220 Old Penitentiary Road Boise, Idaho 83712

Phone: (208)334-2235 Fax: (208)334-2382

Richard F. Hudson, PhD, Chief

EPA Lab ID00018 **CLIA Certified Lab**

Workorder: E080900074 090908

ANALYTICAL RESULTS

Chemistry samples are disposed of after 45 days unless the laboratory is notified otherwise. Lab ID: E080900074-007 Date Received: 9/9/2008 Matrix: Water Sample Date Collected: 38 DUP 09/08/2008 00:00 Location: **LAGOON 2** Parameters **Results Units** Report Limit Analyzed By Qual **MCL** -- General Chemistry --Analysis Desc: EPA 300.0 Analytical Method: EPA 300.0 Chloride 50mg/L 10 9/30/2008 F1 Sulfate 28mg/L 10 9/30/2008 $F1$ Analysis Desc: EPA 350.1 Analytical Method: EPA 350.1 Ammonia as N 15mg/L 0.10 9/19/2008 **BO** Analysis Desc: EPA 351.2 Analytical Method: EPA 351.2 **Total Kjeldahl Nitrogen** 26 mg/L 1.0 9/18/2008 **BO** Analysis Desc: EPA 353.2 Analytical Method: EPA 353.2 Nitrogen, Nitrate-Nitrite 0.026 mg/L 0.010 9/24/2008 **BO** Analysis Desc: EPA 365.1 Analytical Method: EPA 365.1 **Total Phosphorus** $5.9_mQ/L$ 0.10 9/29/2008 EI Analysis Desc: SM 2540C Analytical Method: SM 2540C **Total Dissolved Solids** 400mg/L 10 9/17/2008 **SH**

Report ID: 24517 - 303971

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EPA Lab ID00018 **CLIA** Certified Lab

Workorder: E080900074 090908

ANALYTICAL RESULTS

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EPA Lab ID00018 CLIA Certified Lab

Workorder: E080900074 090908

ANALYTICAL RESULTS

Chemistry samples are disposed of after 45 days unless the laboratory is notified otherwise. Date Received: 9/9/2008 Lab ID: E080900074-009 Matrix: Water Date Collected: 09/08/2008 00:00 Sample 41 Location: **I PONDE** Report Limit Analyzed By Qual **MCL** Parameters **Results Units** -- Metals --Analysis Desc: EPA 200.7 Preparation Method: EPA 180.1 Analytical Method: EPA 200.7 Calcium 19mg/L 0.10 9/24/2008 **JS JS** Magnesium 7.5 mg/L 0.10 9/24/2008 Potassium $5.3_{mq/L}$ 0.10 9/24/2008 **JS JS** Sodium $35_{ma/L}$ 0.10 9/24/2008 -- General Chemistry --Analysis Desc: EPA 300.0 Analytical Method: EPA 300.0 Chloride 33mg/L 2.0 9/30/2008 EI Sulfate 25 mg/L 2.0 9/30/2008 EI Analysis Desc: EPA 350.1 Analytical Method: EPA 350.1 Ammonia as N 0.033 mg/L 0.010 9/19/2008 **BO** Analysis Desc: EPA 351.2 Analytical Method: EPA 351.2 **BO Total Kjeldahl Nitrogen** 0.92 mg/L 0.10 9/18/2008 Analysis Desc: EPA 353.2 Analytical Method: EPA 353.2 2.3 ma/L 0.10 9/24/2008 **BO** Nitrogen, Nitrate-Nitrite Analytical Method: EPA 365.1 Analysis Desc: EPA 365.1 EI **Total Phosphorus** 0.046 mg/L 0.0050 9/22/2008 Analytical Method: SM 2320 B Analysis Desc: SM 2320 B Alkalinity as (CACO3) 88.0mg/L 1.0 9/16/2008 **SM** Analysis Desc: SM 2540C Analytical Method: SM 2540C **Total Dissolved Solids** 180mg/L 10 9/19/2008 **SH**

Report ID: 24517 - 303971

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EPA Lab ID00018 CLIA Certified Lab

Workorder: E080900074 090908

ANALYTICAL RESULTS

Chemistry samples are disposed of after 45 days unless the laboratory is notified otherwise. Lab ID: E080900074-010 Date Received: 9/9/2008 Matrix: Water Sample 43 Date Collected: 09/08/2008 00:00 **I POND W** Location: **MCL Results Units Report Limit Analyzed** Qual Parameters By -- Metals --Analysis Desc: EPA 200.7 Preparation Method: EPA 180.1 Analytical Method: EPA 200.7 9/24/2008 **JS** Calcium 26ma/L 0.10 Magnesium $8.8_{mg/L}$ 0.10 9/24/2008 **JS** Potassium 7.2 mg/L 0.10 9/24/2008 **JS** 9/24/2008 **JS** Sodium 57mg/L 0.10 -- General Chemistry --Analysis Desc: EPA 300.0 Analytical Method: EPA 300.0 Chloride 44mg/L 10 9/30/2008 EI EI Sulfate 44mg/L 10 9/30/2008 Analytical Method: EPA 350.1 Analysis Desc: EPA 350.1 0.022 mg/L **BO** Ammonia as N 0.010 9/19/2008 Analysis Desc: EPA 351.2 Analytical Method: EPA 351.2 **BO** Total Kjeldahl Nitrogen $3.2_{ma/L}$ 0.10 9/18/2008 Analytical Method: EPA 353.2

 0.11 mg/L

 0.23 mg/L

116mg/L

230mg/L

Analytical Method: EPA 365.1

Analytical Method: SM 2320 B

Analytical Method: SM 2540C

0.010

0.0050

 1.0

10

9/24/2008

9/22/2008

9/16/2008

9/19/2008

BŌ

EI

SM

SH

Analysis Desc: EPA 353.2 Nitrogen, Nitrate-Nitrite

Analysis Desc: EPA 365.1

Analysis Desc: SM 2320 B

Analysis Desc: SM 2540C

Alkalinity as (CACO3)

Total Dissolved Solids

Total Phosphorus

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2220 Old Penitentiary Road Boise, Idaho 83712 Phone: (208)334-2235 Fax: (208)334-2382

Richard F. Hudson, PhD, Chief

Matrix: Water

EPA Lab ID00018 **CLIA Certified Lab**

Workorder: E080900074 090908

ANALYTICAL RESULTS

9/9/2008

09/08/2008 00:00

Chemistry samples are disposed of after 45 days unless the laboratory is notified otherwise.

Date Received:

Date Collected:

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2220 Old Penitentiary Road Boise, Idaho 83712 Phone: (208)334-2235 Fax: (208)334-2382

Richard F. Hudson, PhD. Chief

EPA Lab ID00018 **CLIA Certified Lab**

Workorder: E080900074 090908

ANALYTICAL RESULTS

Chemistry samples are disposed of after 45 days unless the laboratory is notified otherwise.

Report ID: 24517 - 303971

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Page 16 of 16

sample color:
Sampling problems: Other observations:

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DEQ Ground Water Sampling Field Sheet

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December 21, 2007

Mr Joe Baldwin Idaho DEQ 1410 N Hilton **Boise ID 83706**

REPORT OF ANALYSES

4 water samples for N, O and H isotopes

C.J.Eastoe **Staff Scientist**

C.L. "BUTCH" OTTER - Governor RICHARD M. ARMSTRONG - Director

Dept. of Env. Quality - State Office Attention: Joe Baldwin 1410 N. Hilton Street

RECEIVED DEC 1 2 2007

IDAHO

DEPARTMENT

HEALTH & WELFARE

DEPT. OF ENVIRONMENTAL QUALITY TECHNICAL SERVICES OFFICE

BUREAU OF LABORATORIES RICHARD F. HUDSON, Ph.D., Chief 2220 Old Penitentiary Road Boise, ID 83712 PHONE 208-334-2235 FAX 208-334-2382

Laboratory Order ID

ΟF

111507 01 (Please refer to this number when contacting the lab)

Boise, ID 83706-1255

Customer Sample ID: $\mathbf{1}$

Lab Sample Number: 0711 0276

Get your forms on the web at: http://www.healthy.idaho.gov "Labs Submission Forms"

Laboratory Supervisor

EPA Laboratory ID: ID00018

Page 1

Dept. of Env. Quality - State Office

EXPRESS THE STREAM ENT OF

C.L. "BUTCH" OTTER - Governor RICHARD M. ARMSTRONG - Director

BUREAU OF LABORATORIES RICHARD F. HUDSON, Ph.D., Chief 2220 Old Penilentiary Road **Boise, ID 83712** PHONE 208-334-2235 FAX 208-334-2382

Laboratory Order ID

111507 01

(Please refer to this number when contacting the lab)

Boise, ID 83706-1255

Attention: Joe Baldwin 1410 N. Hilton Street

Customer Sample ID: 2

Lab Sample Number: 0711 0277

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Laboratory Supervisor

EPA Laboratory ID: ID00018

Page 1

C.L. 'BUTCH' OTTER - Governor **RICHARD M. ARMSTRONG - Director**

Dept. of Env. Quality - State Office Attention: Joe Baldwin 1410 N. Hilton Street

BUREAU OF LABORATORIES RICHARD F. HUDSON, Ph.D., Chief 2220 Old Penilentary Road Boise, ID 83712 PHONE 208-334-2235 FAX 208-334-2382

Laboratory Order ID

111507 01 (Please refer to this number when contacting the lab)

Boise, ID 83706-1255

Customer Sample ID: 4

11/14/2007 **Date Collected: Time Collected:** 12:50 PM **Date/Time Received:** 11/14/2007 **Collected By: Tressa Nicholas**

Lab Sample Number: 0711 0278

Get your forms on the web at: http://www.healthy.idaho.gov "Labs Submission Forms"

Laboratory Supervisor

EPA Laboratory ID: ID00018

Page 1

C.L. "BUTCH" OTTER - Governor RICHARD M. ARMSTRONG - Director

Dept. of Env. Quality - State Office Attention: Joe Baldwin 1410 N. Hilton Street

BUREAU OF LABORATORIES RICHARD F. HUDSON, Ph.D., Chief 2220 Old Penitentiary Road Boise, ID 83712 PHONE 208-334-2235 FAX 208-334-7382

Laboratory Order ID

111507 01 (Please refer to this number when contacting the lab)

Boise, ID 83706-1255

IDAHO DEPARTMENT OF

Customer Sample ID: 5

Lab Sample Number: 0711 0279

Get your forms on the web at: http://www.healthy.idaho.gov "Labs Submission Forms"

Laboratory Supervisor

EPA Laboratory ID: ID00018

Page 1

Mr Joe Baldwin **December 21, 2007**

Idaho DEQ 1410 N Hilton Boise ID 83706

REPORT OF ANALYSES

4 water samples for N, O and H isotopes

C.J.Eastoe Staff Scientist

