

Sampling Plan

for

Detection of septic system waste in the groundwater
of Beaumont CA using chemical and isotopic tracers

University of California, Riverside
SWRCB Agreement No. R8-2010-0022

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1. GENERAL APPROACH

This document establishes the sampling plan for CAL EPA STATE WATER RESOURCES CONTROL BOARD (SWRCB) Agency Award No: R8-2010-0022: “Detection of septic system waste in the groundwater of Beaumont CA using chemical and isotopic tracers”. Septic systems may be a threat to groundwater quality in the Beaumont, CA area. Septic systems contain a large suite of inorganic and organic substances, some of which have only recently been recognized as having negative effects on human health and the environment. These emerging contaminants can be extremely toxic at low levels and produce effects on the endocrine systems of higher organisms. In the proposed study, samples will be collected from groundwater wells in and around the City of Beaumont CA, in a synoptic survey. Additional samples of surface water in the region (urban and natural streams, agricultural drainage and groundwater recharge basins) will be co-collected to examine water quality in waters recharging the aquifer. The surface and groundwater samples will be analyzed to determine concentrations of chemical and isotopic constituents that are diagnostic of the presence of septic wastewater in groundwater. These constituents include major cations (Ca, Mg, Na, K, B), major anions (Cl, SO₄, Br), dissolved organic carbon (DOC), nutrients (NH₄, NO₃, total N, dissolved organic N), isotopes of nitrate ($\delta^{15}\text{N}$ and $\delta^{18}\text{O}$) and emerging pharmaceutical, pesticide, and food additive contaminants. Using these diagnostic tracers and results from modeling of groundwater movement using MODFLOW 96, the investigators will assess the threat of septic systems to groundwater quality in the study region.

2. SAMPLING SITES

2.1 Wells

Well water sampling locations were chosen from a list of 54 Key Well Water Quality Program Wells that were previously sampled within the Beaumont Management Zone (Wildermuth 2010; Table 1 and Figure 1). Using a list of contacts supplied by Dr. Cindy Li of the SWRCB and assistance from Samantha Adams (Senior Scientist II, Wildermuth Environmental, Inc.) we attempted to contact the owner/operators of all publically operated wells and a subset of privately operated wells (Table 1). Table 1 and Figure 1 show all 54 Key Program Wells and are colored coded as to the wells’ sampling status: a) green wells will be sampled, b) pink wells are no longer operated and cannot be sampled, c) red wells will likely be

sampled, but permission to access is still being negotiated and d) black wells are duplicative of other sampled wells and we do not intend to sample them except at the direction of SWRCB. The total number of wells to be sampled will be between 30 and 40 and they provide excellent spatial coverage of the Beaumont Management Zone and encompass all major landuse types (Figure 1).

Additional surface water sampling sites have been identified and are listed in Table 2. These sites include the BCVWD Recharge Basin, Little San Gorgonio Creek (2 locations), and Smith Creek (2 locations). The creek sampling sites are positioned so that we can collect runoff that is: a) predominantly from upland areas with little urban influence and b) near the creeks' intersection with Interstate 10 with greater influence from non-point urban contaminant sources. The two creeks will be sampled on two dates to provide a better picture of surface water quality over the course of the rainy season: (a) a first flush storm in the early part of the rainy season and (b) during a large rain event in January or February (weather permitting). The BCVWD Recharge Basin utilizes State Water Project water and local runoff in Little San Gorgonio Creek. We will coordinate our sampling of the BCVWD Recharge Basin to coincide with periods when only Project water is being used for recharge; we plan to collect two samples of the BCVWM Recharge Basin between November 2010 and February 2011.

Proper interpretation of the stable isotope values of nitrate, requires understanding of end-member isotopic composition. While broad patterns in $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$ value exist (Kendall 1998), site-specific end-member values for nitrate provide additional information that can be used in mixing models to determine groundwater nitrate sources. To better understand inputs of nitrate to groundwaters of Beaumont from septic tanks and agricultural operations, we will attempt to acquire: (a) septic fluids from local Septic Pumping companies and (b) collect surface run-off originating on poultry ranches during winter rain events (Table 3).

Table 1. Well sampling sites. Permission has been granted to sample wells shown in green. Wells shown in purple have discontinued operations according to well owners/operators. Sites in red are high priority wells for sampling and permission is currently being negotiated. Sites in black text are duplicative and are not planned to be sampled.

Well Number	Well Owner	UTM Easting	Longitude Northing
0	Almo, M.C.	501832.318	3753005.698
1	Beaumont Cemetery	503380.900	3753003.040
2	Beaumont Cherry Valley Water District	502723.670	3755313.088
3	Beaumont Cherry Valley Water District	502920.506	3755311.762
4	Beaumont Cherry Valley Water District	502907.868	3755205.032
5	Beaumont Cherry Valley Water District	503224.512	3760400.012
6	Beaumont Cherry Valley Water District	503553.772	3760837.162
7	Beaumont Cherry Valley Water District	503430.870	3763011.388
8	Beaumont Cherry Valley Water District	504647.326	3765017.622
9	Beaumont Cherry Valley Water District	504860.197	3765116.043
10	Beaumont Cherry Valley Water District	504997.261	3765248.651
11	Beaumont Cherry Valley Water District	504253.537	3764826.190
12	Beaumont Cherry Valley Water District	502912.902	3759076.975
13	Beaumont Cherry Valley Water District	504627.699	3764993.106
14	Beaumont Cherry Valley Water District	504717.412	3764973.949
15	Beaumont Cherry Valley Water District	504666.443	3764986.073
16	Beaumont Cherry Valley Water District	503293.078	3757562.700
17	Beaumont Cherry Valley Water District	502885.314	3756094.850
18	Beaumont Cherry Valley Water District	502490.000	3757737.000
19	Beaumont Cherry Valley Water District	500220.570	3757664.560
20	Beaumont Cherry Valley Water District	504151.000	3756122.000
21	Beaumont Cherry Valley Water District	503441.000	3755078.000
22	Beaumont Cherry Valley Water District	498511.704	3758423.000
23	Britton, Larry	498494.000	3757277.000
24	California Oak Valley Golf And Resort LLC	500681.000	3756648.000
25	California Oak Valley Golf And Resort LLC	500336.560	3756839.650
26	Cherry Valley Mutual Water Co.	501814.000	3760073.000
27	Cherry Valley Nursery	500095.000	3758396.000
28	Cherry Valley Water Company	500029.000	3758878.000
29	Desert Lawn Funeral Home and Memorial	498094.000	3757277.000
30	Dowling, Francis M	500416.660	3754357.150
31	Downing, Randy	498331.900	3759376.900
32	East Valley Golf Club	497356.000	3756555.000
33	East Valley Golf Club	498779.000	3756714.000
34	Illy, Stefan	500978.278	3759867.107
35	MCM Poultry	499544.000	3753783.000
36	Oak Valley Partners	495985.000	3758865.000
37	Oak Valley Partners	495346.086	3759044.000

Well Number	Well Owner	UTM Easting	Longitude Northing
38	Oak Valley Partners	495356.000	3759175.000
39	Pistilli, Joe	501474.810	3753148.460
40	Rogers, Mildred	495966.000	3758959.000
41	Sharondale Mesa Owners Association	496103.000	3759672.000
42	Sharondale Mesa Owners Association	496254.000	3759672.000
43	South Mesa Water Company	494872.179	3761434.054
44	South Mesa Water Company	495036.451	3760833.490
45	South Mesa Water Company	493697.850	3761616.040
46	Sunny-Cal Egg & Poultry Company	498792.970	3758454.620
47	United States, Geological Survey	502543.000	3759307.000
48	United States, Geological Survey	502545.000	3759306.000
49	United States, Geological Survey	502447.670	3759426.510
50	United States, Geological Survey	502447.670	3759426.510
51	United States, Geological Survey	502447.670	3759426.510
52	Yucaipa Valley Water District	495460.071	3760181.469
53	Yucaipa Valley Water District	495785.965	3759731.055

Table 2. Surface water sampling sites.

Site Number	Well Owner	UTM Easting	Longitude Northing
1	Beaumont Cherry Valley Water District Recharge Facility	502258.000	3758489.000
2	Little San Gorgonio Creek @ Orchard Street	502497.000	3759496.000
3	Little San Gorgonio Creek @ Oak Valley PKWY	500764.000	3756255.000
4	Smith Creek near Highland Springs Resort	505415.000	3758476.000
5	Smith Creek near @ I-10	507150.000	3753860.000

Table 3. Isotope end-member sampling.

Site Number	Well Owner	UTM Easting	Longitude Northing
1	MCM Poultry	499544.000	3753783.000
2	Sunny-Cal Egg & Poultry Company	498792.970	3758454.620
3	Wright Septic Tank Services	Beaumont,	California
4	Patrick's Septic Tank Services	Yucaipa	California
5	Honest John's Septic Services Inc.	Apple Valley	California

3. SAMPLING METHOD REQUIREMENTS

Well samples will be collected from hose bibs or alternate devices at each sampling location. Water will be drained from the pressure tank with the well pump turned off. Once empty, the pump will be turned on and the pressure tank will be allowed to refill. These steps will be repeated once more. The water will then be turned on and remain on throughout the remainder of the sampling procedure. A 5 gallon bucket will be filled from a valve nearest the wellhead, and the time will be recorded. Water pH, temperature, conductivity, and dissolved oxygen (DO) will be measured using a YSI meter and probe to ensure water is representative of local groundwater. These probes will be rinsed with well water and then used to measure parameters until the values obtained stabilize over a pre-determined time interval. These values are shown in Table 4. If there is any treatment system attached to the well, water will be sample prior to treatment, or the well may have to be removed from the sampling list if pre-treated water sampling is not possible.

Table 4. Well water flushing stability criteria.

Field Measurement	Stability Criteria ¹
pH	± 0.3 standard units
Temperature (T)	± 0.4°C (Thermistor thermometer)
	± 0.8°C (liquid-in-glass thermometer)
Conductivity ($\mu\text{S cm}^{-1}$ at 25°C)	± 1.0% for $\text{SC} \leq 100 \mu\text{S cm}^{-1}$
	± 0.5% for $\text{SC} > 100 \mu\text{S cm}^{-1}$
Dissolved Oxygen (mg L^{-1})	± 0.5 mg L^{-1}

¹Allowable variation between 3 or more sequential field measurement values taken every 5 minutes

Creek samples will be collected by dipping sample bottles underwater with a gloved hand or by using a telescoping sampling pole to hold the sample bottle for dipping into the creek. All sample bottles will be rinsed 3x with sample before filling. A small sampling pump with tubing will be used to collect septic fluid and surface runoff from poultry ranches. Every 10th major ion sample from well, creeks and surface waters will be collected in duplicate.

Samples for major anions, cations, nutrients and stable isotope measurements will be collected after filtering through a 0.45 μm pore size, Whatmann Polycap GW capsule filter. Major cation and anion samples will be collected in a new 1-liter HDPE bottle that has been soaked in deionized water (18 megaohm) for several days and rinsed three times with filtered sample water; these samples will be stored at 5°C. Samples for nutrient fractions will be collected in a new 0.25-liter HDPE bottle that has been soaked in deionized water (18 megaohm) for several days and rinsed three times with filtered sample water; these samples will be filled to the neck and stored frozen at -20°C. Filtered samples for DOC will collected in 40 ml amber bottles and preserved with 2 drops of trace metal grade HCl. Samples for organic SWI analyses

will be collected in pre-cleaned glass sample bottles that are triple rinsed with sample. Organic SWI sample collection bottles will be cleaned with laboratory detergent, a brush, and hot tap water. After scrubbing, the glass bottles will be rinsed three to four times with de-ionized water and either burned at 400 °C for four hours or rinsed consecutively with 5 ml MTBE, 5 ml HPLC grade methanol and 5 ml ultra-pure water. The glassware will then be stored with dedicated glassware to prevent external contamination of samples. The SWI samples will not be field-filtered and instead will be transported by the laboratory on ice for furthering processing. At the lab the SWI samples will be filtered and extracted within 48 hours of sampling. Every 10th SWI well sample will be collected in duplicate.

3.1 Sample Handling and Custody Requirements

Samples are labeled with individual site codes, sample date and time, and numbered consecutively starting with number one. The result is a unique identification combination for each sample collected. These identification labels are also entered directly on to the chain-of-custody form in the field. A sample chain of custody (COC) will accompany every sample taken in the field. An example of the COC is presented in Appendix A of the QAPP. Immediately following collection, samples will be kept on ice in a cooler until they are delivered to the UCR laboratory. The UCR laboratory keeps a copy of the chain-of-custody form.

4. CHEMICAL ANALYSES

A variety of inorganic (Table 5), isotopic (Table 5) and organic (Table 6) analyses will be conducted during our groundwater investigation. The chemical species to be determined were chosen to: (a) provide diagnostic indicators of the presence of septic wastewater in groundwater (e.g., NO₃, NH₄, DON, DOC, isotopes of NO₃, emerging contaminants), (b) to help identify groundwater flow patterns and potential pollutant sources (e.g., major cations and anions) and (c) provide general knowledge of groundwater conditions in the Beaumont region (e.g., pH, alkalinity, specific conductance, Cl and SO₄). Note: quality assurance and control procedures for all chemical analyses are contained in the project QAPP document which accompanies this Workplan.

4.1 Major Ions, Nutrients and Isotopes of Nitrate

Specific conductance measurements will be made with a laboratory conductivity meter equipped with a graphite conductivity electrode with a cell constant of K=1 cm⁻¹. Laboratory pH will be made with a high quality, laboratory pH meter equipped with an Orion-Ross combination electrode. The pH meter and electrode will be calibrated using pH buffer solutions (4, 7 and 10) and the calibration checked by measuring the pH of two weak-HCl solutions (10⁻⁴ N (pH: 4.0)

and 10^{-5} N (pH: 5.0)). Acid neutralizing capacity of samples will be measured by Gran Titration using the calibrated laboratory pH meter and Ross electrode. Hydrochloric acid with a normality of 0.1 will be used to titrate the sample past the equivalence point. At least four titrant-pH measurement pairs will be made between pH 4.3 and 3.7 and used in the Gran computation.

Major anions (Cl, Br, NO₃, NO₂ and SO₄) will be measured using chemically suppressed ion chromatography on a Dionex ion chromatograph following EPA Method 300.1 or a modification thereof. Major cations will be measured by inductively coupled plasma - atomic emission spectroscopy (EPA Method 200.7).

Ammonium will be measured using the phenol-hypochlorite method (modified EPA 350.1). Total dissolved nitrogen (TDN) will be analyzed in samples after NaOH-potassium persulfate digestion, with the nitrate produced by the digestion measured by EPA Method 353.2. Dissolved organic nitrogen will be computed as the difference between TDN and dissolved inorganic nitrogen (nitrate+nitrite+ammonium). Dissolved organic carbon will be measured on a Shimadzu TOC 5050 employing high-temperature Pt-catalyzed combustion (EPA 9060A).

Isotopes of nitrate will be measured using the microbial denitrifier method of the USGS Reston Laboratory (RSIL Code 2900). In this method, bacteria (*Pseudomonas chlororaphis* and/or *P. aureofaciens*) are used to convert NO₃ into N₂O gases which are then led to an isotope ratio mass spectrometer which measures the $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$ of the N₂O and through computational means, arrives at the $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$ of the NO₃ in the samples. Analyses will be performed at the Facility for Isotope Ratio Mass Spectrometry at UC Riverside.

Table 5. Summary of general water quality and isotopic constituents to be measured in water samples collected during the Beaumont investigation.

Analyte	Well Samples	Creek Samples	Poultry Runoff	Septic Fluids	Analytical Method
pH, Alkalinity	X	X	X		pH Electrode and Meter
Specific Conductance	X	X	X		Meter and K=1 cm ⁻¹ cell
Major Anions (Cl, Br, NO ₃ , SO ₄)	X	X	X		EPA Method 300.1
Major Cations (Ca, Mg, Na, K, B)	X	X	X		EPA Method 200.7

Nutrients (NH ₄ , Total Dissolved N, organic N)	X	X	X		EPA Method 350.1, NaOH-Persulfate digestion, EPA 353.2
Dissolved Organic Carbon	X	X	X		EPA Method 9060A
δ¹⁵N and δ¹⁸O of Nitrate	X	X	X	X	USGS Method (RSIL Lab Code 2900)

4.2 Emerging Contaminants

The analysis of the organic septic waste indicators (SWI) in water is based on the methods developed by Vanderford and Snyder (2006). In method development, rigorous procedures will be used to validate the recovery, precision, and determine the instrument limits of detection (ILOD). The compounds of interest are shown in Table 6. SWI analyses will be conducted on all well and creek samples and in samples from the BCVWD recharge facility. The use of UPLC-MS/MS to detect SWI in water includes several steps. These steps are preparation of stock and working solutions of the target compounds and their labeled counterparts, sample preparation, sample extraction, instrument calibration and QA/QC evaluation.

Standard Preparation: SWI compounds and their isotopically labeled counterparts will be purchased from Sigma Aldrich (St. Louis, MO), Toronto Research Chemicals (North York, Ontario, Canada), United States Pharmacopeia (Rockville, MD), and C/D/N Isotopes, Inc. (Pointe-Claire, Quebec, Canada). Individual stock solutions (100 µg L⁻¹ or 10 µg L⁻¹ for each compound) will be prepared by weighing the exact amount of each compound and dissolving in methanol. A multiple SWI working solution (100 ng L⁻¹ of each compound) will be prepared by appropriate dilutions of the stock solutions in methanol.

UPLC-MS/MS Analysis: Analysis will be conducted using Aquity UPLC system coupled with a Trinity triple quadrupole mass spectrometer equipped with an electrospray ionization source (ESI) (Waters, Milford, MA). The column will be a BEH C18 column (100mm X 2.1mm i.d. with 1.7 µm particle size). Individual tune files will be created by infusing the individual compounds to determine the optimum capillary and cone voltages, collision energies, product ions. The ILOD ranges from 0.1 to 10 ng/ml for individual analytes, for the listed SWIs.

Table 6. Emerging contaminants to be used as indicators of septic contamination of groundwater in the Beaumont region.

Compound	Use	Compound	Use
17 α -Ethinylestradiol	Oral Contraceptive	Estrone	Human Hormone
Acetaminophen	Analgesic	Fluoxetine	Anti-depressant
Atenolol	Beta Blocker	Gemfibrozil	Fibrate
Atorvastatin	Statin	Ibuprofen	Non-steroidal Anti-inflammatory
Bisphenol	Plasticizer	Meprobamate	Human Tranquilizer
Caffeine	Stimulant	Naproxen	Non-steroidal Anti-inflammatory
Carbamazepine	Anti-epileptic	Primidone	Anti-convulsant
DEET	Insect Repellent	Simvastatin	Statin
Diazepam	Barbiturate	Sulfamethoxazole	Antibiotic
Diclofenac	Non-steroidal Anti-inflammatory	TCEP	Flame Retardant
Dilantin	Anti-epileptic	Triclosan	Antibacterial
Diuron	Herbicide	Trimethoprim	Antibiotic

5. GROUNDWATER MODELING

The objectives of the modeling component of this project are two-fold: (a) assess the aquifer vulnerability to potential contamination from septic systems existing under different hydrogeological and meteorological conditions and (b) Estimate the potential recharge zones for the different ground water wells being analyzed for contamination.

We will assess the aquifer vulnerability in the study area using a variety of statistical and numerical approaches. Ground water vulnerability maps are designed to show areas of greatest potential for ground-water contamination on the basis of hydrogeological and anthropogenic (human) factors. The maps are developed by using computer mapping hardware and software called a geographic information system (GIS) to combine data layers such as land use, soils, and depth to water. Usually, ground-water vulnerability is determined by assigning point ratings to the individual data layers and then adding the point ratings together when those layers are combined into a vulnerability map. An example of a simple vulnerability assessment method is DRASTIC, named for the seven factors considered in the method: Depth to water, net Recharge, Aquifer media, Soil media, Topography, Impact of vadose zone media, and hydraulic Conductivity of the aquifer (Twarakavi and Kaluarachchi, 2005). We will also analyze the applicability of statistical approaches such as logistic regression to assess ground water vulnerability in the area (Twarakavi and Kaluarachchi, 2005; 2006a; 2006b). Use of these statistical methods helps in identifying regions of high risk with the model domain.

We will also use HYDRUS-1D (Simunek et al., 2010) to numerically assess the vulnerability of the aquifer to contamination from septic systems. HYDRUS-1D uses the Richard's equation for water flow and convection-dispersion equation for the solute transport through the unsaturated zone. We will use HYDRUS-1D to assess the movement of solutes through soils to ground water. HYDRUS-1D gives information about the potential contaminants in the unsaturated zone as well as the ability of the soils to leach the contaminants to ground water. Together with the information provided by HYDRUS-1D model and holistic-based ground water vulnerability methods, we will summarize the susceptibility of the aquifer to contamination.

Our second objective is to estimate the extent of recharge zones for each of the ground water wells under consideration. We will accomplish this objective using MODFLOW. MODFLOW is

a three dimensional finite-difference ground water model that simulates steady and non-steady flow in complex aquifer systems subjected to various external stresses such as flow to wells, areal recharge, evapotranspiration, flow to drains, and flow through river beds. The estimated recharge zones for each of the wells would be analyzed together with aforementioned modeling efforts as well as contamination data obtained from the field for outlining the state of quality of ground water resources in the study area.

6. PROJECT SCHEDULE

The project officially began on July 1, 2010 (Table 7). During the initial project period, existing reports on groundwater in the Beaumont region were reviewed (Wildermuth 2010, Rewis et al., 2006) and this Workplan was developed in collaboration with Dr. Cindy Li and Samantha Adams (Task 1). Groundwater sampling will begin in November and continue into January (Task 2). Because the holding times for emerging contaminants and nutrients are short, we cannot collect and process more than 5 to 10 samples per sampling trip, thus we anticipate 2 sampling trips per month at ca. biweekly intervals. Creek water samples and surface runoff from poultry ranches will be collected during the first large rain event of the rainy season – this usually occurs in December. Another creek water and surface runoff sampling will take place during a large rain event, weather permitting, sometime in the winter (January through March 2011). We also anticipate sampling the BCVWD recharge facility in early 2011 when SWP deliveries are being made. Sampling from septic pumping trucks will be accomplished during the scheduled biweekly well sampling trips.

Chemical analyses of well water, creek water, surface runoff and septic fluids will begin in November 2010 and be completed by the end of June 2011 (Tasks 3, 4 and 5). Groundwater modeling will begin January 2011 once all hydrogeological input data are acquired from resource agencies such as the USGS. We anticipate that the modeling work will continue through the end of August 2011 given that all chemical analyses will be completed in June 2011.

Progress reports will be prepared and submitted to Dr. Cindy Li on a quarterly basis (Table 7). This Workplan contains information on the progress of the study to date and is submitted as evidence for completion of Task 1 and, thus, it serves as our first quarterly report. A final report, summarizing all project findings and recommendations will be completed by December 1, 2011 and submitted to the SWRCB.

Table 7. Project schedule and timeline of Tasks.

Start Date: July 1, 2010. End Date December 1, 2011

	Jul-Sep '10	Oct-Dec '10	Jan-Mar '11	Apr-Jun '11	Jul-Aug '11	Oct-Dec- '11
Task 1: Workplan development	X	X				
Task 2: Water sampling		X	X			
Task 3: Inorganic analyses		X	X	X		
Task 4: Isotope analyses			X	X		
Task 5: Organic analyses		X	X	X		
Task 6: Modeling.			X	X	X	
Task 7: Reports						
Quarterly:		10/31/2010	1/31/2011	4/30/2011	7/31/2011	10/31/2011
Final:						12/1/2011
Task 8 Project Management	X	X	X	X	X	X

7. LITERATURE CITED

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Figure 1. Land use map of Beaumont with all potential sampling sites identified.

