Guidelines for Sampling and Analyzing Solutions from Aquifer Thermal Energy Storage Systems

September 1982

Prepared for the U.S. Department of Energy
Office of Energy Systems Research
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
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BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

Printed in the United States of America
Available from
National Technical Information Service
United States Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151

NTIS Price Codes
Microfiche A01

Printed Copy

Pages	Price Codes
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026-050	A03
051-075	A04
076-100	A05
101-125	A06
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GUIDELINES FOR SAMPLING AND ANALYZING
SOLUTIONS FROM AQUIFER THERMAL
ENERGY STORAGE SYSTEMS

W. J. Deutsch

September 1982

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the U.S. Department of Energy
under Contract DE-AC06-76RL0 1830

Pacific Northwest Laboratory
Richland, Washington 99352

FOREWORD

The seasonal thermal energy storage (STES) concept involves storing thermal energy, from such sources as winter chill, summer heat, and industrial waste heat, for future use in heating and cooling buildings or industrial processes. Widespread development and implementation of STES would significantly reduce the need to generate primary energy in the U.S.

Research efforts in STES under Pacific Northwest Laboratory management encompass aquifer thermal energy storage (ATES) and STES technology assessment and development. Aquifers represent a logical thermal storage medium because of their natural capacity for retaining heat and cold. Aquifer thermal energy storage has the potential to significantly conserve and/or displace relatively scarce and costly petroleum fuels using near-term technology.

Research in ATES has been aimed toward better characterizing system performance. To successfully store and retrieve heat or chill using an aquifer medium, the entire system must be compatible with the natural ground-water system. Thus, ground-water composition at any ATES site must be thoroughly understood. This paper presents guidelines for sampling and analyzing ground-water solutions to provide the type and extent of data necessary for successful ATES system operation.

J. R. Raymond
Program Manager
Aquifer Thermal Energy Storage Program

SUMMARY

The successful aquifer storage and retrieval of energy in the form of heated or chilled water require that the engineered system be compatible with the natural ground-water system. The composition of the ground water must be well known to predict and avoid potential problems that heating or cooling the water may create for operation of the plant. This paper presents a set of guidelines for sampling and analyzing solutions from simulated or real ground-water energy storage systems.

Sampling guidelines include methods for flushing wells of stagnant water and monitoring selective solution parameters (pH, Eh, temperature or conductivity) as indicators of the efficiency of flushing. Certain unstable ground-water parameters (temperature, pH, Eh, dissolved oxygen, and conductivity) should be measured onsite. It is recommended that alkalinity, sulfide, and ammonia determinations be done within 24 hr of sampling. In addition to these field measurements, samples of the ground water should be filtered, preserved, and stored for laboratory analysis of major cations, anions, trace metals, organic and inorganic carbon and certain redox couples ($\text{Fe}^{2+}/\text{Fe}^{3+}$ and $\text{As}^{3+}/\text{As}^{5+}$). The final results of the analysis should be verified by computing the cation-anion balance and comparing measured conductivity with the solution analysis.

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GUIDELINES FOR SAMPLING AND ANALYZING SOLUTIONS FROM AQUIFER THERMAL ENERGY STORAGE SYSTEMS

INTRODUCTION

The storage of thermal energy in aquifers is being studied at Pacific Northwest Laboratory (PNL) as part of the Seasonal Thermal Energy Storage (STES) Project. In a typical storage plan, ground water is pumped from wells, raised in temperature by surplus industrial heat, and reinjected into the aquifer for storage and seasonal energy needs. The heating, aeration, and subsequent cooling of the water may plug the aquifer, degrade water quality, and promote corrosion and/or plugging of the surface plant and downhole equipment. These potential engineering and water-quality problems are being examined in field and laboratory studies of aquifer storage systems. The chemical analysis of ground-water samples from STES sites (an important part of these studies) helps explain the processes that occur when thermally altered water is injected and extracted from a STES system. Procedures are given in this paper for collecting complete and accurate chemical and related physical data on STES systems.

General guidelines for collecting, preserving, and analyzing water samples can be found in standard references published by the American Public Health Association (Greenberg, Connors and Jenkins 1980), the Environmental Protection Agency (EPA 1979), and the U.S. Geological Survey (Brown, Skougstad and Fishman 1970; Skougstad et al. 1979). Field methods for measuring selected unstable constituents (temperature, pH, Eh, alkalinity, dissolved oxygen, and conductivity) are described by Wood (1976). Presser and Barnes (1974) and Watson (1978) discuss special techniques for analyzing geothermal waters, while Kennedy, Zellweger and Jones (1974) present compelling evidence that the solution must be properly filtered in the field to remove clay-size sediments prior to sample preservation or analysis. These references and practical field and laboratory experience form the basis for the procedures that follow.

PROCEDURES

A thorough analysis of ground-water composition requires collecting data on the parameters listed in Table 1. The complete set of parameters will not have to be measured for each water sample, but must be measured for a representative number of samples to provide an accurate chemical characterization of the aquifer storage system. This section describes the sampling and analysis procedures to be used.

SAMPLING TECHNIQUES

Samples of ground water from wells can be taken by a variety of techniques, including pumping, bailing, and downhole grab sampling with remotely-triggered containers. The goal in using these techniques is to obtain, with minimum difficulty, a sample of ground water that represents in-situ chemical conditions. In general, wells will have to be flushed of standing water that has been contaminated by contact with the well casing and atmosphere. During flushing, unstable parameters of the ground water (e.g., pH, Eh, temperature) can be monitored to determine when flushing is complete and native ground water can be sampled (Humenick, Turk and Colchin 1980). A well can be flushed by either bailing or pumping; however, pumping is the preferred method because of its relatively high flushing rate and because it allows for continuous well-head monitoring of the solution's pH, Eh, and temperature using established techniques (Potter et al. 1979; Wanty, Langmuir and Chatham 1981). Gibb, Schuller and Griffin (1981) recommend that peristaltic or submersible diaphragm-type pumps be used and pump tests be run on the wells prior to sampling to determine sampling frequency and pumping rate and duration. In general, the pumping rate during flushing should be kept low (<20 L/min) to reduce turbulence in the well and possible contamination from the atmosphere. The water chemistry in most small-diameter (<10 cm), shallow (<100 m) wells will stabilize in less than 30 minutes at this pumping rate.

TABLE 1. Important Solution Parameters

Physical Parameters		
pH	Temperature	Total dissolved solids
Eh	Specific conductivity	
Common Constituents		
Ammonium	Dissolved carbon dioxide	Nitrate
Bicarbonate	Dissolved inorganic carbon	Potassium
Calcium	Dissolved organic carbon	Silicon
Carbonate	Dissolved oxygen	Sodium
Chloride	Magnesium	Sulfate
Trace and Minor Elements		
Aluminum	Iron (total dissolved)	Phosphate
Arsenic (total)	Iron (II,III)	Selenium
Arsenic (III, V)	Lead	Silver
Barium	Lithium	Strontium
Boron	Manganese	Sulfide
Chromium	Mercury	Uranium
Cobalt	Molybdenum	Vanadium
Copper	Nickel	Zinc
Fluoride	Nitrite	

If pumping and concurrent monitoring of a well are not feasible, then bailing or downhole grab sampling will be required. Both of these techniques involve time-consuming and repetitious sampling to determine if the diagnostic contamination parameters have stabilized and a representative sample can be collected. Bailing and grab sampling techniques are not recommended for routine sampling.

Field studies of ground-water chemistry and water/rock interactions can be augmented by laboratory experiments using dynamic (flow loop) or static (reaction vessel) equipment. Sampling protocol for laboratory simulations does not require extensive pretesting prior to solution sampling because the

system should be free of contaminants. Samples can be taken soon after bleeding off the small amount of solution that may have stagnated near the sampling port.

SAMPLE COLLECTION AND PRESERVATION

After the monitored ground-water parameters have stabilized, a set of water samples can be collected. The samples should be of a certain volume and should be preserved in accordance with the techniques listed in Table 2. Measurements of pH, Eh, temperature, conductivity, and dissolved oxygen—if they are to be performed onsite—should be taken when each sample is collected. Suggested methods for these measurements and for alkalinity, sulfide, and ammonia analyses, which must be done within 24 hr of sampling, are listed in the Field Determinations section. The remaining measurements of cations, anions, organic and inorganic carbon, $\text{Fe}^{2+}/\text{Fe}^{3+}$, and $\text{As}^{3+}/\text{As}^{5+}$ can be performed within the specified time limits (Table 2) on preserved samples. These measurements are described under Laboratory Analyses.

As shown in Figure 1, most samples should be filtered before they are preserved and stored or analyzed (Kennedy, Zellweger and Jones 1974). Filtering is not performed if measurements must be made immediately or if the sample would be adversely influenced by the filtering process (e.g., loss of CO_2 gas). Samples are filtered to remove particulates that may bias or interfere with the solution analysis. A filter pore size of 0.22 microns or smaller should be used. If possible, the samples should be pressure-filtered rather than vacuum-filtered to preclude the loss of volatiles and reduce the possibility of changes in sample volume due to evaporation. Samples that require acidification should have enough high-purity acid added to lower pH to ~1.5. Samples for sulfide measurements must be stabilized with an antioxidant buffer, and arsenic samples must be frozen. It has been shown that both the arsenic (Cherry et al. 1979) and iron (Nordstrom, Jenne and Ball 1979) redox couples can be used as indicators of the oxidation state of natural water; consequently, their measurement will be used in selected cases to verify Eh measurements by the platinum electrode.

TABLE 2. Sample Size Requirements and Preservation Techniques

Measurement	Volume (ml)	Preservative	Holding Time
pH	100 ml(a)	Det. on site	None
Eh	100 ml(a)	Det. on site	None
Temperature	100 ml(a)	Det. on site	None
Conductivity	100 ml(a)	Det. on site	None
Dissolved oxygen	300 ml	Det. on site	None
Cations (major elements and trace metals)	25 ml(b)	HNO ₃ to pH<2 Cool, 4°C	14 days(c)
Anions (SO ₄ , NO ₃ , Cl, F, Br)	25 ml(b)	Cool, 4°C	7 days
Organic and Inorganic Carbon	100 ml	Cool, 4°C	7 days
Sulfide	50 ml	Anti-oxidant buffer Cool, 4°C	24 hr
CO ₂ (gas)	125 ml	Cool, 4°C	14 days
Ammonia	100 ml	HNO ₃ to pH<2 Cool, 4°C	24 hr
Alkalinity	200 ml	Cool, 4°C	24 hr
Fe ²⁺ /Fe ³⁺	100 ml	HCl to pH<2 wrap bottle with Al foil, Cool, 4°C	not established
As ³⁺ /As ⁵⁺	100 ml	Freeze	(1 month)(d)

- (a) These sensitive parameters are best measured onsite in a flow-through, bypass system at the well head or in-situ at the intake point. Depending on the technique used, a single 100-ml sample may be sufficient for these four measurements.
- (b) These sample volumes are those required for inductively-coupled plasma and ion chromatographic analysis on the constituents. Larger volumes would be required for alternative techniques.
- (c) Major metal constituents of the water can be preserved by acidification for up to 6 months. However, to accurately measure trace metals, the analysis should be done as soon as possible (Greenberg, Connors and Jenkins 1980).
- (d) Holding time not definitely established, but estimated at a minimum of 1 month.

*SAMPLES STORED AT 4°C

FIGURE 1. Schedule for Completing Analyses

All samples, except for the $\text{CO}_2(\text{gas})$ sample, should be collected in polyethylene containers having polyseal caps; the $\text{CO}_2(\text{gas})$ sample container should be made of glass. Prior to use, containers should be washed and then rinsed first with 10% nitric acid, followed by a minimum of three washings with distilled, deionized waters. Sample containers should be labeled with a sample number, collection date and time, field treatment, analyses to be performed, and the sampler's name. This information should also be entered in a field/lab notebook. The samples should be protected at all times from contamination or deterioration during transportation.

FIELD DETERMINATIONS

The analyses listed in Table 3 measure ground-water properties that change relatively rapidly due to chemical and biological processes that may occur in the solution after it is removed from its source. Because these properties cannot be adequately preserved using standard techniques, they must be measured immediately upon sampling or within 24 hr, and are best done at the field site.

An in-line sampling cell fed off a bleed from the pump line is the preferred method of measuring pH, Eh, temperature, conductivity, and dissolved oxygen at the surface. This apparatus can serve the dual purpose of monitoring the flushing of the well and measuring unstable properties of the ground water. Alkalinity ($\text{HCO}_3 + \text{CO}_3$), sulfide, and ammonia are measured on individual samples that can be held, with proper preservation, up to 24 hr. Suggested measurement methods are given in Table 3.

LABORATORY ANALYSES

Major cations and anions, trace metals, organic carbon, dissolved CO_2 , and the redox couples $\text{Fe}^{2+}/\text{Fe}^{3+}$ and $\text{As}^{3+}/\text{As}^{5+}$ are determined in the laboratory on preserved samples. Although preservation techniques are believed to stabilize these constituents for a considerable period of time (7 to 28 days), it is recommended that the analyses be performed as soon as possible. The sample volumes required for the cation and anion analyses (25 ml each) assume that sophisticated instrumentation be used for these analyses. Inductively-coupled

TABLE 3. Field Techniques

Measurement	Method
pH	Electrode. standardized and corrected for temperature
Eh (mv)	Platinum electrode, calibrated and corrected for temperature
Temperature (°C)	Standard mercury thermometer
Conductivity (mhos/cm)	Conductivity meter with temperature compensation
Dissolved oxygen (ppm)	Dissolved oxygen probe
Sulfide (mg/l)	Specific ion electrode
Ammonia (mg/l)	Specific ion electrode
Alkalinity (mg/l)	Titration with standardized acid

plasma emission spectroscopy is used for most of the cations (Al, B, Ba, Ca, Cd, Co, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, Si, Sr, Zn); atomic adsorption spectroscopy is used for the remainder (As, Cr, Cu and V). Anions (Br, Cl, F, NO₃, NO₂, PO₄, and SO₄) are determined with an ion chromatograph. Because these analytical techniques are so versatile, they are generally available at most research laboratories. Alternative techniques to measure this suite of cations and anions would require larger sample volumes than those given in Table 2.

Organic and inorganic carbon and dissolved CO₂ analyses can be made with a carbon analyzer that employs an infrared analyzer to detect CO₂ released from a volume of volatilized solution or, in the case of dissolved CO₂, in equilibrium with the water sample. This instrumentation is common in laboratories where organic research is done. As(III) and As(V) can be determined by evolution of arsenic as arsine prior to spectrophotometric analysis as described by Forbeck (1973). Differentiation of As(III) and (V) is accomplished by either 1) measuring As(III) and As(total) and deriving As(V) by difference, or 2) measuring As(III) and then adjusting solution pH to eliminate As(III) followed by direct measurement of As(V). Fe(II) can be determined by complexation with ferrozine (Stookey 1970) or bathophenanthroline (Lee and Stumm 1960; Ghosh, O'Connor and Englebrecht 1967) followed by

colorimetric measurement. Fe(III) is determined by measuring total dissolved iron and Fe(II) and computing the difference between them.

Evaluation of Analytical Data

The results of the field and laboratory measurements can be verified by computing the cation-anion balance and comparing measured conductivity with the solution analysis. The sum of the cations, in milliequivalents per liter (me/l), should equal the sum of the anions. A large positive or negative cation-anion imbalance is indicative of a poor-quality chemical analysis or the lack of analysis for an important constituent. The cation-anion balance (CAB) is calculated using

$$CAB = \frac{\sum \text{Cations (me/l)} - \sum \text{Anions (me/l)}}{\sum \text{Cations (me/l)} + \sum \text{Anions (me/l)}} \times 100\%$$

A 5% tolerance limit is generally considered applicable to studies of groundwater systems. If the imbalance is >5%, the analyses must be repeated.

A fortuitous combination of errors may result in a good cation-anion balance but inaccurate total analysis. Comparing measured conductivity with the analytical results will aid in discovering such discrepancies. With this method, the concentrations of the individual ions are multiplied by appropriate conductivity factors. The sum of the ion conductivity is compared to the measured conductivity to provide an estimate of the completeness and accuracy of the analysis. Complete details of this method are given in Greenberg, Connors and Jenkins (1980).

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