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Downhole Fluid Sampling at the SSSDP California State 2-14 Well Salton Sea, Califomia

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Cover Photograph: Flow line of the SSSDP California State 2-14 well during the flow test in March 1986.

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Edited by Fraser Goff Lisa Shevenell C. O. Grigsby Bert Dennis

Contributors

dy. L. Shevenell P.E. Trujillo, Jr. lell** rger*

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*Sandia National Laboratories, Albuquerque, NM 87115. "US Geological Survey, Menlo Park, CA 94025. tUniversity of California at Berkeley, Berkeley, CA 94720. ^Lawrence Berkeley Laboratory, Berkeley, CA 94720.

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DOWNHOLE FLUID SAMPLING AT THE SSSDP CALIFORNIA STATE 2-14 WELL SALTON SEA, CALIFORNIA

Edited by

Fraser Goff, Lisa Shevenell, C. 0. Grigsby, and Bert Dennis

ABSTRACT

Sea Scientific Drilling Project (SSSDP) well during late December In situ fluid sampling activities were conducted at the Salton 1985 and late March 1986 to obtain unflashed samples of Salton Sea brine. In late December, three sampling runs were made to depths of approximately 1800 m and temperatures of 300°C. In late March, 10 sampling runs were made to depths of approximately 3150 m and temperatures of 350"C. In brief, the Los Alamos tool obtained samples from four of eight runs; the Lawrence Berkeley tool obtained samples from one of one run; the Leutert Instruments, Inc., tool obtained samples from zero of,three runs; and the USGS quartz crystal experiment was lost in the well. The most complete sample was obtained from run #11, using the Los Alamos sampler and Sandia battery pack/controller on a wireline. About 1635 m² of brine, two noble gas samples, and two bulk gas samples were collected from this run. Samples of brine and gas from productive runs have been

distributed to about 15 researchers for various types of analyses. Chemical analyses by the Los Alamos and US Geological Survey analytical teams are presented in this report although they are not corrected for flashing and precipitation.

I. INTRODUCTION

The Salton Sea Scientific Drilling Project (SSSDP) has been designed to study the high-temperature hydrothermal system of the Salton Sea geothermal field, California (Fig. 1). This project is part of the US Continental Scientific Drilling Program (CSDP), a collaborative program funded by the Department of Energy (DOE), the National Science Foundation (NSF), and the US Geological Survey (USGS). After much deliberation, the site of the SSSDP was chosen on state land leased to Kennecott Minerals Co., and the well was officially named the California State 2-14 (Fig. 2; Elders 1985).

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Figure 1. Generalized geologic map of the Salton Trough and adjacent areas showing location of the Salton Sea geothermal field (from Robinson et al. 1976).

AGUNA SALADA

(Dry lake)

The purpose of this report is to summarize the in situ fluid sampling activities that involved the California State 2-14 well from October 1985 to April 1986. Acquisition of unflashed and uncontaminated brine samples under in situ conditions was considered to be an important part of the scientific and technical objectives of the project. During the project a variety of downhole sampling devices were used with seven-conductor and single-conductor cables and a battery pack/wireline to obtain in situ fluids. Because of the high-temperature (350°C) and high-salinity environment (total dissolved solids about 300,000 mg/kg), the performance of these devices was highly variable as discussed below. A gas extraction system was built to extract gas samples from the downhole samplers after decompression and cooling to surface

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Figure 2. Site map of SSSDP California State 2-14 well .

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conditions . Fiel d analytica l procedure s an d samplin g o f th e hypersaline brine s ar e als o described . Finally , th e chemica l analyse s o f th e samples, uncorrected for flashing or precipitation, are listed in the tables. Interpretation of the chemical data will be incorporated into later publications.

II . DESCRIPTIO N O F TOOLS

A . Lo s Alamo s I n Sit u Sampler

ine Los Alamos downhole sampler (Fig. 3) is 8.9 cm (3-1/2 in.) in diameter and is designed to attach to a standard wireline cable head assembly (Archuleta et al. 1978). For the SSSDP project, two new tools were built having 2-^g capacities, and the samplers were made to be compatible with the Sandia battery pack/controller described below. Once the sampler reaches the desired depth, a temperature-hardened electric motor opens a valve to the pre-evacuate d sampl e chamber , an d th e chambe r fill s immediately . Th e motor then reverses direction and closes the valve. Besides use on the Los Alamos Hot Dry Rock project (to 300°C), the in situ sampler has been used successfully in geothermal wells at Valles caldera (232°C), Miravalles, Costa Rica (240°C), and the East Pacific Rise (160° to 290°C). Until the SSSDP project, little attempt was made to quantitatively recover gas from the in situ sampler althoug h ga s sample s wer e collecte d i n bot h th e Valle s calder a an d Miravalles projects.
B. Lawrence Berkeley In Situ Sampler

The Lawrence Berkeley sampler was designed and built for the Gas Research Institute to obtain <u>in situ</u> samples in geopressured wells such as occur on the lexas-Louisiana Gulf Coast (Weres et al. 1984; Solbau et al. 1986). The sampler was designed to be operated to temperatures of 230°C and to contain flui d wit h interna l pressure s u p t o 138 0 bar s (20,00 0 psi) greate r tha n the external pressure. The material of construction is MP35N stainless steel, impartin g grea t strengt h an d corrosio n resistance.

The sample chamber volume is $1 \,$ $\boldsymbol{\textit{k}}$, and the complete sampler tool has an outside diameter of 5.7 cm, length of 3 m, and weight of 55 kg (Fig. 4). Sinker bars can be attached to the sampler to increase its weight. A single conductor cabl e 0.5 5 c m (7/3 2 in.) i n diamete r i s require d t o operat e the sample r whe n i t i s ru n downhole.

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The valves of this flow-through sampler are cocked open at the surface and are kept open by an electromagnet energized by current flowing through the cable. Interrupting the current causes the valves to close irreversibly. All electrical components are immersed in fluorocarbon oil that is in hydrostatic equilibrium with the well bore fluid. This sampler succeeded in bringing up a sample of water from a well in the Geysers geothermal field at a temperature of about 260°C and a pressure of $\sqrt{70}$ bars (1000 psi). Although the condensed steam was recovered, the noncondensable gas was not (T. Box, Freeport Minerals, oral communication, 1986).

The practical temperature limit of the sampler is set by its nonmetallic components, which are the O-rings that serve as static seals, the insulation of the electromagnetic coil, the fluorocarbon oil that protects the electrical subassembly, and the fluorocarbon grease that lubricates the sampler. C. Sandia Battery Pack/Controller

At the request of Wilfred Elders (University of California, Riverside, SSSDP--Principal Investigator), the Los Alamos team became involved in three attempts to sample the SSSDP well with a tool made by Leutert Instruments, Inc., of Houston, Texas. Well-site operations were managed by Agnew and Sweet of Bakersfield, California. The Leutert tool is 4.00 cm (1-37/64 in.) in diameter and about 2 m in length. It is a flow-through tool designed to sample oil field petroleums and brines at temperatures of 150°C or less. The tool has an adapter that can be used to extract associated gas, and the adapter was easily connected to the gas extraction system built by Los Alamos.

The Sandia battery pack was designed to control downhole motors in tools operating in geothermal wells at temperatures in excess of 300°C (Wolfenbarger 1986). The first version of the battery pack was designed specifically to operate the Los Alamos downhole sampler for use in the SSSDP (Fig. 5) . A dewar (vacuum heat shield) houses a battery pack and the electronics used to control the downhole motor in the Los Alamos sampler. Table I shows the design requirements for the battery pack reproduced from Wolfenbarger (1986). In addition, Sandia designed an adapter/bulkhead and seals used to mate the battery pack to the Los Alamos sampler. One of the silver seals used in the adapter/bulkhead could not withstand the severe conditions of the SSSDP well and failed as described below. The problem was finally remedied by welding the sampler and the battery pack together. D. Leutert In Situ Sampler

Figure 5. Photograph of Sandia Battery Pack/Controller (shiny cylinder) connected to the Los Alamos downhole sampler (dull cylinder) in preparation for run #3, December 31, 1985.

TABLE I

DESIGN REQUIREMENT FOR THE SNL BATTERY PACK/CONTROLLER (from Wolfenbarger 1986)

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Valves are mechanically opened at the surface and either closed at the desired depth using a timer clock or jerked closed using a jarhead. Because of the high temperature encountered at SSSDP, this tool failed three times in three attempts. However, Los Alamos has purchased one of these tools and is modifying the seals to adapt the tool for 250°C applications in slim holes. Modification of the tool for use at temperatures above 300°C will require complete redesign using different steels, valves, and closure mechanisms. E. Los Alamos Gas Extraction System

In order to reconstruct the downhole composition of the fluids sampled at Salton Sea, the gas/liquid ratio as well as the gas and liquid compositions must be known. A gas extraction line was designed and constructed to remove the gas from the sampler for analysis and to measure the volume of gas collected. A line drawing of the gas extraction system is presented in Fig. 6. Most of the system is constructed of 0.32-cm (1/8-in.) stainless steel tubing and swage-lock fittings, but vacuum connections and the connections to the gas sample bottle are 0.64-cm (1/4-in.) stainless steel. The actual connection to the gas sample bottle is through flexible silicon tubing so that the sample bottle can be agitated during sample collection. The sample bottle is a modified Giggenbach bottle with a flow-through valve stem (Sheppard and Giggenbach 1985). This bottle is filled approximately 1/3 full (100 ml) with a 4N NaOH solution that absorbs CO_2 , H₂S, and NH₃. Sample bottle preparation is described by Nehring et al. (in prep.) and Sheppard and Giggenbach (1985). Sample ports for collection of helium isotopes in copper tubes were also provided. Details of the helium sampling apparatus are given by Smith and Kennedy (1983). The system also has a coiled tube section that can be placed in a dewar with liquid nitrogen to condense out vacuum oil from the vacuum pump, if desired.

Because the gas pressures are not known in advance, the gas extraction system has a 0-20-bars (0-300-psia) primary pressure gauge and a 0-2-bars (0-30-psia) secondary gauge, which can be isolated from the rest of the system for pressures above 2 bars (30 psia). An expansion chamber is also provided so that system pressures can be adjusted to prevent overpressuring the gas sample bottle. This expansion chamber consists of a smooth cylinder with a gas-tight piston connected to a lead screw. There is a certain amount of play in the connection of the piston to the screw, and if the pressure in the gas

extraction system is above atmospheric pressure, the piston can slide within the chamber and change the internal volume of the system by 4.20 cm^3 .

For practical use the extraction system is divided into several subsystems between valves A, B, C, D, etc. Each subsystem has an associated internal volume, which is accurately measured in order to compute correct volumes and pressures within the sample chamber. The subsystem volumes were measured by a sequence of calibrations wherein a known volume of gas at known pressure was expanded into a given subsystem. The resulting change in pressure was used to calculate a subsystem volume using the ideal gas law. The original "known" volume was a downhole sample bottle whose internal volume was measured by filling the bottle with water and measuring the volume of water. Two sample bottles have a volume of 1985 ml, while the third bottle has a volume of 1987.5 ml. The relative error in this measurement is about 0.25% (<5 m% in 2000 m%) due to gas bubbles and water droplets that cannot be removed on filling or emptying the bottle. Errors in reading the 0-300-psia

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gauge are about 0.1 psi; errors in reading the 0-30-psia gauge are 0.01 psi. For the subsystems measured, the relative error in measuring the volume is approximately 0.25%.

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III. HISTORY OF SAMPLING RUNS A. December 1985 Trip

Run $#2$: For run $#2$ the LANL sample bottle was closed just enough to keep vacuum and to minimize the torque needed by the motor to open the valve. At

This trip lasted from December 27, 1985 to January 2, 1986. On December 29 and 30 surface flow line samples were obtained for comparison with downhole samples. At 10:45 a.m. on December 30, the conditions of the flow line at sampling port #3 were 229° C (445°F) and 17-28 bars (250-400 psi) with surging. At about 2:00 a.m. on December 31, 1985, a measurement by Kuster tool producing horizon of 1866 m (6120 ft) was also 298°C. Several hundred thousand gallons of fluid had been produced from the well before these temperatures were obtained. A summary of sample runs follows and is documented in

yielded an estimated downhole temperature of 298°C at 1814 m (5950 ft). A USGS measurement of temperature using a thermistor probe at the identified Table II.

Run $#1$: At 3:00 a.m. on December 31, 1985, the LANL sampler, which was evacuated and attached to the new USGS TFE-insulated and MP35N-armored sevenconductor cable, started into the hole at 150 ft/min. At depths' below 5000 ft, the resistance between the cable and ground dropped rapidly from over 100,000 « to 20-30,000 «. This drop in resistance effectively leaked power from the cable, and thus it could not provide enough power to adequately operate the electric motor in the downhole sampler. Nonetheless, several attempts were made to open the sampler at 6120 ft. The sampler started the trip up the hole at 4:00 a.m. and reached the surface at 5:30 a.m. When it was taken apart, it contained 108 m² of fluid, which subsequent chemistry showed to be Salton Sea brine. Apparently there was sufficient power to open the valve on the sampler, enough for fluid to leak in, but insufficient power to close the valve. Because of the incomplete seal, gas leaked out of the sampler and pressures could not be maintained.

At 6:00 a.m. members of the drilling and downhole sampling teams conferred and decided to do a very quick rebuild of the USGS Gearhart-Owens cable

head to see if this would fix the power leak.

II

Sampler empty; power loss along cable was very severe. A decision was made to
try to open sampler at lower temperature, higher in well. Sampler opened but
would not close tightly due to low power. Thus, sample is flashed. No sample collected Complete fluid and gas aampler empty; sampler would not open effectively due to power loss along
cable and/or leaky cable head. Valve opened enough, or leaked, to let some
Fluid in. No qas was collected. Sampler empty; battery pack fails to open sampler but a cracked inlet valve
on sampler allows some fluid to leak in. No qas collected. f ail. fireline breaks after roughly 24 h in well. Experimental tool falls to
octom of well. Fishing operations fail. No sample collected. ϵ Complete fluid sample collected but gas is fails to open 1
solution is
sion. No sample collected. and seal between battery pack and sampler
No sample collected, **GEOTHERMAL** reason. Sampler partly filled with 283 mL O.1N HNO₃; sampler
unknown reason. No sample collected but nitric acid
collected to study tool resistance to internal corros sample collected. Sampler empty; sampler opens and closes perfectly.
samples collected. $2 - 14$ and seal fail again. STATE f or Sampler empty; sampler fails to open ϵ Seals fail. No sample collected. OPERATIONS AT THE SSSDP CALIFORNIA
WELL, SALTON SEA, CALIFORNIA No sample collected Seals fail; clock timer leaks. wampler closes perfectly.
Iost for unknown reason. Sampler empty; connector
Battery pack blows fuse. empty, connector Seals fail. Sampler TABLE II Sample
No. $55 - 7;$
 $55 - 8$ $5 - 23$ $S - 21$ $5s - 22$ $s-s$ $5 - 6$ Volume
Sample
(m&) $\tilde{\epsilon}$ \mathbf{r} 316 1635 813 4^{2505} $351^{\rm c}$ 351° 351° 351° $351^{\rm c}$ $351^{\rm c}$ $\overline{\begin{smallmatrix} 1\text{ emp} & \cdots \end{smallmatrix}}$ 298^{a} $298^{\mathbf{a}}$ 151° 351 ^c 351 ^C 351° Kuster tool measurement at 2:00 AM, December 31, 1985,
Estimated temperature.
Kuster tool measurement at 9:00 PM, March 22, 1986. OF DOWNHOLE SAMPLING $\frac{Dent}{(m)}$ 1511 3110 866 1866 $\frac{3171}{2}$ $\overline{111}$ $\overline{3171}$ $\overline{3171}$ 3171 $\frac{3110}{2}$ 3110 3110 3171 USGS Quartz Crystal
Experiment on Otis Wireline Los Alamos Sampler-Sandia
Battery Pack-Otis Wireline Lawrence Berkeley Sampler-
USGS 1-Cond. Cable Los Alamos Sampler-Sandia
Battery Pack-Otis Wireline ampling Trip #2 (March 21 to March 26, 1986) 1 12/31/85 2:00 A - Los Alamos Sampler-USGS
5:30 A 7-Cond. Cable Los Alamos Sampler-USGS
7-Cond. Cable Leutert Sampler-Agnew &
Sweet Wireline Leutert Sampler-Agnew &
Sweet Wireline Sampling Trip #1 (Dec. 29, 1985 to Jan. 1, 1 Leutert Sampler-Agnew
Sweet Wireline Configuration $\begin{bmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{bmatrix}$ SUMMARY $4:30 P -$
 $8:30 P$ $2:30 P -$
 $3:30 P$ $4:30 P-$
5:30 P $1:00 A-$
 $3:30 A$ $4:30 A -$
 $8:30 A$ $5:30 A -$
 $8:00 A$ $10:30A-$
 $1:30P$ $2 12/31/85$ 7:30 A-
10:30A $11:30P-$
 $3:00A$ $12:30A -$
 $1:30P$ $10:00P -$
 $10:00P$ $3:00 P -$
 $6:30 P$ Time 12/31/85 Date 6 3/23/86 5 3/23/86 12 3/25/86 7 3/23/86 8 3/23/86 $0.3/25/86$ 11 3/25/86 13 3/25/86 4 3/22-
23/86 $93/23-24/86$ $\n ^R$ un

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7:30 a.m. the tool went back into the well at 150 ft/min on the USGS cable. At 4957 ft the tool was stopped because the resistance in the cable had dropped to $10-12,000$ Ω . It was obvious that the resistance would drop even more at the higher temperature at 6120 ft, so a decision was made to try to open the sampler at this depth to get a sample in the well below the flashing level. According to the LANL instrumentation, the sampler opened and closed although a weak response was noted on the instruments. At 8:30 a.m. the sampler was tripped out of the hole and reached the surface at 10:00 a.m. When the tool was taken apart, visible rust coated the inlet valve of the sampler. This valve was not tightly closed, and therefore no gas pressure was maintained. About 316 m% of fluid was in the sampler, and this brine contained a considerable amount of precipitated material. Apparently the motor did not receive enough power to close the valve, and the sample flashed continuously all the way to the surface.

Because the LANL sampler-USGS cable configuration was not successful. it was decided to use the Sandia battery pack instead of the USGS cable for any future sampling runs. The USGS cable was rigged down at 11:30 a.m., and the Otis wireline began to rig up at 1:30 p.m.

Run $#3$: At 2:00 p.m. Sandia began to prepare the battery pack and to hook it to the LANL sampler. At 4:30 p.m. the sampler/battery pack combo (Fig. 5) was lifted to the rig floor, and the internal clock of the battery pack was activated to open at approximately 6:50 p.m., 2 h, 20 min after setting. At 5:00 p.m. the combo started into the hole at 80 ft/min and returned to the surface at $8:00$ p.m. By $8:30$ p.m. the sampler was connected to the gas extraction system, but the measured internal pressure at 52°C was only 1.76 psia, meaning that the sampler was empty or near empty. Samples of this gas were collected for noble gas and bulk gas analyses. The sampler was then opened and only 15 m of fluid was inside. A 1- m split of this sample was given to Bill Carothers (USGS) to determine conductivity.

It was then noticed that the inlet valve was plugged with salt (NaCl). It appeared that salt had precipitated from the brine and plugged the inlet port of the sampler when the brine flashed into the evacuated chamber. A modification to the sampler was proposed and begun in preparation of a fourth run using the second Los Alamos sampler, but Sandia personnel announced that the battery pack was shorted out. When the standby battery pack was prepared

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for use, another short circuit was created. Thus, at 10:00 p.m. on December 31, 1985, in situ sampling activities of the first sampling trip ceased.

Analysis of Too! Performance, Run #3: The sampler was taken apart and examined in the laboratory. At that time a longitudinal crack was found in the inlet needle valve, but the motor that opened and closed this valve was found to be operational. It was concluded that, rather than salt plugging the inlet port of the sampler, the silver O-ring seal between the sampler and the battery pack had failed, allowing brine to leak into the wires connecting the motor and battery pack. The hot brine then corroded the wires causing the battery pack to short-circuit. The fluid inside the sample chamber merely leaked in through the cracked needle valve.

In preparation for the second sampling trip in March, the Los Alamos tools were cleaned and new valves obtained. Sandia changed their circuitry to accommodate possible short circuits due to overloading conditions. The design of the metal seal in the bulkhead connecting the two tools was not modified even though there was doubt that the design was adequate. However, it was decided to use only the battery pack on all future sampling runs because of the very poor power delivery of the USGS cable in the severe conditions of the Salton Sea brine. An attempt was made to get Lawrence Berkeley Laboratory to use their sampler during at least one sampling run. Also, W. Elders informed F. Goff that Agnew and Sweet would run yet another in situ sampler, built by Leutert Instruments, Inc.

preparation for attaching the sample bottles to the gas extraction system, a leak was noted in the bulkhead connecting the sampler and battery pack. The measured internal pressure of the sampler was 1.19 psia at 58°C, indicating that little or no fluid was in the sampler. When the sampler was opened, it contained no fluid.

B. March 1986 Trip

The March trip lasted from March 19 through 27, 1986. Surface flow samples were collected for comparative purposes on March 21, and geophysical logs were run on March 22. About one million gallons of brine flowed from the well on March 21. The frothy nature of the brine indicated that it contained considerable diesel and drilling-mud contaminants. The main flow zone to be sampled was apparently at depths below 3080 m (10,100 ft). At 9:00 p.m. on March 22, the Kuster tool indicated a temperature of 351°C at approximately this depth.

Run #4: At 10:30 p.m., March 22, 1986, the Los Alamos sampler/Sandia battery pack combo was lowered into the SSSDP well on the Otis wireline at about 100 ft/min to a depth of 10,400 ft. The sampling combo was left at this depth for about 1/2 h; it arrived back at the surface at 3:00 a.m. on March 2 3. While the sampler and the battery pack were being broken apart in

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An examination of the bulkhead connecting the two tools revealed that the silver O-ring seal had leaked and brine had corroded the wires. Thus, no power was delivered to the motor from the battery pack and the sampler did not open. Welding the bulkhead to the sampler may have remedied the situation, but because of the time (4:00 a.m.), it was decided to make another run with a new seal and to pack the bulkhead interior with heavy grease to try to keep the brine out.

Run #5: The sampler/battery pack combo went back into the well at about 5:00 a.m. to the same depth as before and returned to the surface at 8:20 a.m. After the gas extraction system was connected to the sample bottle, an internal pressure of 1.04 psia was measured at 52°C, indicating little or no fluid was inside. When the chamber was opened, no fluid was in it. Examination of the bulkhead and seal showed that the grease had been decomposed by the high-temperature brine that had leaked inside. The brine literally cooked the connecting wires into a decomposed mess $(Fiq, 7)$. No more runs were planned with the sampler/battery pack combo until bulkhead repairs could be made.

Run #6: While modifications were being made to the sampler/battery pack bulkhead and seal, the Otis wireline was rigged down, and Agnew and Sweet rigged up to run the Leutert downhole sampler. During these procedures, W. Elders asked F. Goff to oversee handling of the fluid samples if the Leutert tool was successful. At 12:30 a.m. the Leutert tool went into the hole using the jarhead trip mechanism, traveled to a depth of 10,400 ft, and returned to the surface at 1:30 p.m. The gas extraction system was connected, but when the pressure was measured, it was about 14.5 psia, indicating the sampler was at atmospheric pressure. When the tool was opened, the sample chamber was empty. The best diagnosis was that the severe temperature had degraded the seals and springs of the sampler.

Figure 7. Photograph of "cooked" connecting wires in the bulkhead built by Sandia to connect the battery pack and sampler. The sealing design failed during runs 4 and 5 and probably during run 3. This difficulty was overcome by welding the joint together.

Run $#7$: At 2:30 p.m. the Leutert sampler again went into the hole to the same depth using the clock-timer trip mechanism and returned to the surface at 3:30 p.m. The operator of the tool (C. Wells, Leutert Instruments) noticed that the valves were not tripped and no sample was inside. When he opened the clock-timer mechanism, a jet of steam burst into the air and several milliliters of Salton Sea brine drained on the ground. The brine had apparently entered the mechanism through failed seals, destroying the clock and, hence, preventing closure of the valves.

Run $#8:$ At $4:30 \times p$ m. a third run was made into the SSSDP hole to a depth of 10,400 ft using the Leutert sampler with jarhead trip mechanism. The tool returned to the surface at 5:30 p.m., and the tool operator noticed

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immediately that the valves had failed to close properly. No sample was contained within the sample chamber. Evidently, the severe temperature had again destroyed the seals and springs, causing the tool to fail.

A decision was made at this point to rig down Agnew and Sweet's wireline and rig up the Otis wireline in preparation for running the USGS Quartz

Crystal Experiment.

Run #9 (USGS Quartz Crystal Experiment): This run is described because it involves fluid sampling and occurred during the other fluid sampling activities. The experiment was devised by Phil Bethke of the USGS, Reston, Virginia. In brief, crushed high-purity quartz crystals are enclosed in a housing that allows surrounding fluids to enter and permeate the crystals. In theory the brine is saturated with silica and will crystallize new quartz from solution onto the seed crystals. Fluid inclusions of brine are trapped during the crystallization process and can be analyzed later in the laboratory.

The USGS Quartz Crystal Experiment went into the SSSDP well at approximately 10:00 p.m. on March 23, 1986, and was left for approximately 24 h at a depth of 10,400 ft. The experiment was completed at about 10:00 p.m. the following day, but when the wireline came to the surface, it had separated, just above its connection, from the housing containing the experiment. Examination of the broken end of the wireline suggested that corrosion during the 24 h in the well had contributed to mechanical failure of the wireline. It was not known precisely when and how the wireline had separated, whether at total depth or during running out of the hole. Because of the relatively small size of the housing (about 0.5 m long and 12 cm in diameter), the experiment presumably fell to the bottom of the well (about 10,600 ft). A decision was made to try to fish the tool from the well, but these operations failed and fishing ceased at about 12:30 a.m. on March 25, 1986.

Run #10: At 1:00 a.m. on March 25, 1986, the Los Alamos sampler/Sandia battery pack was run into the SSSDP well to a depth of 10,200 ft. The bulkhead and seal unit joining the sampler and battery pack was welded to the first joint of the sampler. In addition, a hole was drilled and tapped in the bulkhead area for filling the connection with silicon oil. The hole was closed with a standard pipe plug. The combo arrived back at the surface at 3:30 a.m., but when the sampler was connected to the gas extraction system, an internal pressure of only 0.75 psia was measured at 55° C, indicating that the

sampler contained little or no brine. The opened sample chamber contained no fluid. The tools were examined, but the reason they failed remained unknown.

Run #11: Another sample chamber and motor unit were assembled, and the chamber was evacuated. At 5:30 a.m. this sampler/battery pack combo was run into the well on the Otis wireline to 10,200 ft and returned to the surface at about 8:00 a.m.

The gas-extracting system was attached to the sample chamber, and at 8:50 a.m. on March 25, 1986, an internal pressure of 51 psia was measured at 60°C. Two bulk gas samples and two noble gas samples were extracted from the sampler by 9:40 a.m. The sampler was opened, and about 1635 m ^{ℓ} of green syrupy liquid was poured out for field measurements and for sample splitting. The sample from Run #11 is the best sample from all attempts to collect in situ fluids from the SSSDP well. Later measurements by the USGS indicated that the conditions at 10,200 ft were about 353°C at 4,287 psia.

Run $#12$: Another Los Alamos sampler was prepared by adding 285 ml of $0.1N$ HNO₃ and pumping to near vacuum (0.10 psia). The sampler/battery pack combo went back into the well at 10:30 a.m. and returned to the surface at about 1:30 p.m. The internal pressure of the sample chamber was 2.55 psia at about 50°C, suggesting that little or no fluid had entered the sampler to mix with the nitric acid. The opened chamber contained about 250 m of chrome-red solution, indicating that the sampler had not opened. A pronounced reaction zone was noted inside the sample chamber where the hot nitric acid solution had rested in the chamber bottom. Examination of the battery pack showed that the batteries still had current, but a loose connection was noted in the circuitry (S. Petty, oral commun. 1986). Subsequent examination of the battery pack at Los Alamos showed that a wire to the motor had been severed during reassembly of this tool before the sampling run.

At about 2:00 p.m. the Otis wireline was rigged down, and the USGS single conductor cable was rigged up in preparation for running in with the Lawrence Berkeley Laboratory (LBL) sampler.

Run #13: At 3:00 p.m. the LBL sampler was run into the SSSDP well on the USGS single conductor cable. At 5800 ft on the way downhole, the weight indicator on the winch failed. When the desired depth of 10,200 ft was reached, the tool's solenoid trip mechanism was activated from the surface, and the tool started back up the hole at about 4:30 p.m. On the way out of the well, the cable wound improperly on the drum, so an extra hour was spent

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rewinding the cable and coming out of the hole very slowly. During the delay the fluid contained in the sample chamber may have had sufficient time to react with metals in the sampler at the elevated temperatures, thus altering its chemistry.

At about 6:30 p.m. the LBL sampler reached the surface and was attached to the gas extraction system. An internal pressure of about 14.4 psia was measured at 50°C, suggesting that gas had leaked from the sampler and had equilibrated with atmospheric pressure. Repeated use of the gas extraction system to pull gas from the sampler yielded the same pressure every time. The gas extraction system was disconnected, and an N₂ gas cylinder was attached to the gas outlet valve of the sample chamber. At a pressure of only 5 psig, some fluid was noted leaking from a joint near the bottom end of the sampler. The N_2 gas was shut off, and an attempt was made to open the valve to drain the fluid. The valve would not open under normal conditions. The tool was then dismantled at the bottom end to gain direct access to the valve. When it was opened, about 813 m% of Salton Sea brine drained suddenly through the valve into a tared beaker. Field analyses were then made and samples were spl it.

No further attempts were made to take in situ samples after run #13. Operations ceased at about 9:00 p.m. on March 25, 1986.

Analysis of Tool Performance, March 1986 Trip: The major problem associated with sampling events of the March trip was failure of the silver O-ring seal in the adapter/bulkhead connecting the Los Alamos and Sandia tools. Other problems were caused by wear and tear from repeated runs into the hostile environment and the complicated process of connecting the battery pack and sampler. This latter problem resulted in the severed wire that may have caused run #12 to fail.

The Leutert tool failed at the SSSDP well because it was not designed for temperatures exceeding 150°C, and the previously mentioned alterations required to improve performance had not been done. The USGS Quartz Crystal Experiment failed because the housing connecting it to the wireline was poorly designed. Probably this design would have been adequate for experiments in environments other than the Salton Sea brine.

The LBL tool was not originally designed for temperatures above 300°C, but it had been modified for the SSSDP project (Solbau et al. 1986). Failure

of this tool to prevent internal gas from leaking, as well as failure of the lower valve to open properly, was probably due to problems associated with the USGS single-conductor wireline winch. Because the cable wrapped on the drum improperly, the tool stayed in the well over an hour longer than expected. This could have caused degradation of seals and valves.

Otherwise, the battery pack concept appears to be promising for extremely hot, hostile geothermal environments where cables cannot deliver large amounts of electric power. Los Alamos recently took one of the Sandia battery packs to Miravalles, Costa Rica (September 1986), and successfully retrieved a downhole sample from an acid well at 240°C. Because of the acid environment, no attempt was made to use the seven-conductor cable available on site.

The LANL downhole sampler has already proved itself a reliable tool in typical geothermal environments (TDS <40,000 mg/kg) at temperatures <300°C where it can be operated from a seven-conductor cable. The tool will probably perform reasonably well to temperatures of 350°C in similar fluid environments without major redesign. In the hot, hypersaline environment of the SSSDP well, the tool could not be effectively operated from a cable because of the high power requirement of the dc motor in the tool and the severe power losses in the cable and/or cablehead. By using the Sandia battery pack/controller, this problem was overcome. However, the design of the battery pack electronics and its seal must be improved before this tool will become reliable.

After the downhole sample was collected and the sampling tool returned to the surface, the sample bottle was removed from the tool and was cooled to below 70°C to lower the water vapor pressure in the bottle and prevent loss of steam during gas extraction. The bottle was then connected to the gas extraction line. Following evacuation of the gas extraction system up to the downhole sample bottle valve, the various subsystems were isolated by closing all valves. The procedure for removing the gas sample is as follows (refer to Fig. 6):

The LBL downhole sampler was much easier to operate at the SSSDP well than the LANL sampler because its closure mechanism requires that much less power be delivered through the single-conductor cable. However, this tool could not retain the gas in the in situ fluids and was very difficult to open. These problems may have occurred only because the tool remained in the well too long, but it would appear that some design changes could alleviate these problems in future work.

IV. SURFACE SAMPLING PROCEDURES

The procedures described below were originally devised in the laboratory but continued to evolve during field sampling at the well site. Gas sampling, fluid analysis, and fluid preservation and splitting are the surface sampling procedures associated with in situ sampling.

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A. Gas Sampling

As described above, the LANL gas extraction system was designed to measure the internal pressure in the tool's sample chamber by releasing the gas pressure into an accurately known volume. The internal volume of the sampler(s) including valves must also be known to correct for the exact internal pressure. After the first pressure measurement is made, the gas inside the extraction system can be "pushed" over to sampling ports using valves and the piston cylinder (see Fig. 6) . When the ports are opened and gas samples are extracted, the pressure of the gas samples can be measured. For the SSSDP operation three sampling ports were provided: two ports for noble gas samples taken by B. M. Kennedy (UC Berkeley) and one port for bulk gas chemistry and light stable isotopes (C. Janik and A. Truesdell, USGS). Because only run #11 produced a "full" sample bottle with no leaks, the best gas samples are presumably from this run. Two noble gas samples collected in copper tubes and two bulk gas samples collected in evacuated glass bottles filled with 4N NaOH were obtained from run #11. In addition, low-pressure gas samples were obtained from runs #3 and #13.

1. Slowly open the sample bottle valve and record the pressure on the

- 0-300-psi gauge.
- 2. Close the sample bottle valve.
-
-
- 5. Open valve B and collect He sample.
- 6. Open valve A and collect gas sample.
- gauge.
- 8. Close valves C and D.
-

3. Open valve D and record pressure on the 0-300-psi gauge. 4. Open valve C and record the pressure on the 0-30-psi gauge. 7. Close valves A and B and record residual pressure in system on 0-30-psi

9. Repeat steps 1-4 and 6-8 until gas sample bottle is filled.

The gas sampling procedures are relatively complicated and require at least two people who can operate the valves, the piston cylinder, the sampling ports, the sample containers, and the recording of data.

B. Field Analysis

Because any high-temperature geothermal fluid is unstable at surface conditions, some routine field measurements must be run immediately after the fluid comes out of the in situ container. This is particularly true for Salton Sea brine, which is highly supersaturated and has high concentrations of reduced iron that rapidly oxidize in contact with air.

All fluids from a sampling run were first drained into a tared $4-\ell$ plastic beaker, and the total weight of liquid was measured on a top-loading balance. Liquid density was next measured by pipetting a $25-m$ aliquot of brine into a tared specific gravity bottle and weighing on the top-loading balance. Total liquid volume within the sample chamber can be computed from the total weight of the fluid and its density. The same 25-ml aliquot is used to measure field pH, field Eh, and dissolved H₂S by means of portable meters and spectrometers.

A separate 30-mL aliquot was pipetted into a plastic beaker, stripped of $CO₂$ by bubbling N₂ gas through the fluid and titrating with $0.1N$ HCl to determine dissolved HCO₃. No other field measurements were made.

C. Field Preservation and Splitting

Before coming to the well site, an elaborate set of guides for sample splitting and preservation was prepared to handle the in situ liquid samples. Due to the difficulty of obtaining samples in the SSSDP well and because many geochemists asked for in situ samples at the site, the original guides were largely ignored to preserve the limited volume of samples in the greatest variety of ways.

A number of diluting methods were tried to prevent Salton Sea brine from precipitating. Probably the best method of preservation is to dilute three parts brine with one part $0.1N HNO₃$. We observed no oxidation or precipitation of brine in the field using this recipe, and $HNO₃$ is a useful acid to keep trace elements in solution. However, upon returning to the laboratory, even nitric-acid-diluted samples will precipitate, requiring an elaborate analytical process to obtain an accurate analysis. Another diluting recipe would be to use O.IN HCl, but some metal chlorides are insoluble, and addition of this acid ruins the sample for Cl analysis. Using concentrated acids for

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dilution causes instant oxidation and precipitation primarily of Mn-compounds. Using distilled water for dilution does not prevent slow oxidation or precipitation of Fe/Mn compounds, or precipitation of silica.

Preserving raw brine will not prevent rapid oxidation and copious precipitation of Fe/Mn compounds, or precipitation of silica and chloride salts. Precipitation of these major compounds removes trace metals from solution by absorption as well as precipitation. Table III lists all samples, their method of preservation if any, and the researchers who received sample splits at the well site. Table IV lists all samples that were sent to researchers after in situ operations were concluded. This inventory is accurate up to September 1986.

V. PRELIMINARY BRINE AND GAS CHEMISTRY A. ESS-1 Laboratory, Los Alamos

Preliminary analyses of samples are listed in Table V. These analyses are not corrected for precipitation, which eventually occurred in most sample bottles. Table V also includes analyses of surface samples collected during flow tests.

In general, the liquid volumes of each sample were measured accurately, and all precipitates were collected on tared filter papers for separate analysis. Because of the high concentrations of salts in the brine, sample aliquots were generally diluted with deionized water for analysis. A detailed description of analytical methods used in this laboratory can be found in Trujillo et al . (1987). Each analysis listed in Table V has been corrected for density and dilution with preservatives and is true at 25°C. Corrected values that include effects of precipitation and flashing (if any) will be given in a later report. B. INC-7 Laboratory, Los Alamos

Sample splits were received and small aliquots from each sample were diluted with deionized water. Samples were analyzed by dc plasma emission spectroscopy and were corrected for density and dilution but not precipitation (Table V) .

C. US Geological Survey Laboratory, Menlo Park

Gas samples extracted from the downhole samplers were analyzed by C. J. Janik at the US Geological Survey, Menlo Park, California, using methods described in Sheppard and Truesdell (1985) and Nehring et al. (in prep.). The

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TABLE III

INVENTORY OF FLUID AND GAS SAMPLES COLLECTED DURING THE IN SITU SAMPLING OPERATIONS AT THE SSSDP WELL, DECEMBER 31, 1985 AND MARCH 25, 1986

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TABLE III (continued)

 $\mathcal{A}=\mathcal{A}$.

UCR - University of California at Riverside UCB - University of California at Berkeley

BNW - Battelle Northwest

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INVENTORY OF FLUID SAMPLES DISTRIBUTED TO RESEARCHERS AFTER THE IN SITU SAMPLING OPERATIONS (CURRENT AS OF SEPTEMBER 1986)

LANL - Los Alamos National Laboratory

UNM – University of New Mexico
USGS – US Geological Survey
LBL – Lawrence Berkeley Laboratory
CIW – Carneqie Institution of Washington
BRGM – Bureau de Recherches Géologiques et Minières (France)
FMC – Freeport Mineral

TABLE V-A

CONDITIONS AND PHYSICAL PROPERTIES OF SELECTED SAMPLES OF BRINE FROM CALIFORNIA STATE 2-14 WELL ANALYZED FOR CHEMISTRY

a
Pensities from sampling trip #1 computed in laboratory at 25°C; densities from sampling trip #2 measured in field at temperature of pH measurement.

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pH and Eh measurements from sampling trip #1 analyzed in laboratory; pH and Eh measurements from sampling trip #2 analyzed in field.

pH of 5.30 measured in field by Carothers (USGS).

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TABLE V-B

CHEMICAL ANALYSES (UNCORRECTED FOR FLASH AND POSSIBLE PRECIPITATION) OF SELECTED SAMPLES (IN mg/kg)
Analyses by P. E. Trujillo, Jr., and D. Counce (LANL)

^a Summation of cations and anions based on Tables V-B and V-C.

b pH value is an estimate only; lab pH is given in Table V-A.

c Bicarbonate titration by Carothers (USGS) in field.

 d Carbonate = 700 mg/kg.

e Bicarbonate titration in field.

TABLE V-C

TRACE ELEMENT ANALYSES (UNCORRECTED FOR FLASH AND POSSIBLE PRECIPITATION) OF SELECTED SAMPLES (IN mg/kg)
Analyses by P. E. Trujillo, Jr., and D. Counce (LANL)

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Sampling Trip #2 (March 21 to March 26, 1986)

TABLE V-D

PRECIPITATION) FLASH AND POSSIBLE FOR (UNCORRECTED
TED SAMPLES (
/ G. K. Bayhu \mathcal{S} . ANALYSES
OF SELEC
Analyses b CHEMICAL ADDITIONAL

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GAS ANALYSES OF DOWNHOLE SAMPLES (IN mol%) Analyses by C. J. Janik (USGS)

aDoes not include about 2% of unidentified organic gas believed to be derived from thermal breakdown of diesel fuel added to drilling fluids.

preliminary results in Table V-E have not been corrected for air contamination. The high concentration of $0₂$ in sample SS-23 indicates that this sample is largely air and, therefore, cannot be used to reconstruct downhole fluid compositions. The analysis of SS-21 does not seem to be 100% due to the presence of an unknown gas comprising approximately 9% of the gases insoluble in the NaOH solution. This gas was detected independently (but not identified) by Bill Evans (USGS) and may be a breakdown product of the drilling fluid, which contained diesel fuel.

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TABLE V-E

supporting and assisting our field activities. Don Michels advised us on how to handle Salton Sea brine samples. Wilf Elders provided support through the entire project from proposal to reporting stages and inspired us during our several attempts to retrieve samples. This project was funded by the US Department of Energy, Office of Basic Energy Sciences, Division of Geothermal and Hydropower Technology.

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