

FIELD EVALUATION OF SAMPLING METHODS FOR  
PRESSURIZED GEOTHERMAL LIQUIDS, GASES,  
AND SUSPENDED SOLIDS

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## ABSTRACT

Many different sampling methods were tested and compared for collecting samples for measurement of brine chemistry, gases, and suspended solids from pressurized geothermal systems. The tests were conducted on the 6-2 wellhead and a test loop at the Department of Energy's Geothermal Test Facility at East Mesa, California. The recommended methods for single-phase liquid or single-phase steam (with gases) are presented, together with detailed procedures. The results of testing methods for sampling two phase liquid-steam systems showed significant errors can result. It was recommended that two-phase flowing wells be directed to a full flow separator and the single-phase liquid and single-phase steam sampled separately using the recommended methods.

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## SUMMARY AND CONCLUSIONS

D. W. Shannon

During this program, sampling methods have been developed for use on single-phase water or steam lines on above-ground geothermal piping systems. When carefully applied, using proper equipment, these methods provide precise and accurate data on most of the chemical species of interest in geothermal energy development. We find, in using these methods, that a cation-anion charge-balance ratio and a sum-of-analyses, total dissolved solids (TDS) mass-balance ratio of 0.95 to 1.0 is routinely obtained. Precision is typically better than +10%.

### PROBE/DOUBLE-COIL COOLING METHOD

The sampling methods are based on inserting a sampling probe into the pipe or vessel of interest and collecting the sample from the main flow. Tapping into the main flow minimizes wall effects or contamination and scale present in the system entry valve (see Figure 3.2). The sample is cooled while still at full system pressure in order to prevent steam flashing from occurring. Ball valves are used on the inlet to preclude a large drop in pressure. After cooling and after the sample is depressurized, gases often break out of solution. These gases are recombined with the liquid in absorption sample collection bottles.

Cooling is rapid enough that no significant silica deposition occurs. However, further work should be done on possible losses of heavy metals and suspended solids in the double cooling coils.

The first coil is immersed in water which boils to extract most of the heat. This is followed by a second coil in an ice water bath that cools the sample to 25°C. By using the heat of vaporization of the cooling water in the first bucket and the heat of fusion of ice in the second bucket, a large cooling system is not necessary. Taking one complete set of samples typically requires the use of 5 gallons of water and 75 pounds of crushed or cubed ice. After flow through the coils has reached steady-state with a constant outlet temperature of 25°C ± 5°C, samples are collected.

Each sample "set" taken from a single sampling point consists of up to seven different bottles, a weighed filter, gas bulb sample and several field measurements. These are summarized in Table S.1. The sample stabilization procedures were adapted from those used by the U.S. Geological Survey.<sup>(1)</sup>

Each group of samples is coded for identification purposes (see Table S.2). The seven bottles are usually separated when they arrive at Pacific Northwest Laboratory so that all the chloride titrations, for example, can be done at one time for cost efficiency. This numbering system readily permits the data to be consolidated again in the data report and for computer storage. The number also allows the scientific personnel familiar with the "test" and "location" numbers to quickly identify pieces of data.

#### Measurement of pH

The pH of geothermal fluids is usually dominated by the  $\text{CO}_2\text{-HCO}_3^{-1}\text{-CO}_2^{-2}$  equilibria. When samples are depressurized,  $\text{CO}_2$  is lost. For this reason, pH should be measured in the field. An in-line, fully pressurized cell at known temperature provides the best pH measurement. For high pressure systems, however, such cells are expensive and difficult to use. In the absence of such a cell, the water and gas from the coil outlet can be directed into a small beaker containing pH electrodes, and a measurement can be made at 1 atmosphere and 25°C. If this is done, the measurement should be reported as a "1 atm" sample at 25°C. The designation alerts readers to adjust their data using  $\text{CO}_2$  and  $\text{HCO}_3^-$  totals to reflect the pH levels of the high temperature brine.

#### Measurement of Total $\text{CO}_2$

The water and gas mixture from the coil outlet was directed into a bottle of 500 ml of absorbing solution (2N NaOH) through a sintered glass frit dispersion tube. Of the seven different methods we tested, this simple, single sparge-bottle method provided the most consistent data. A single sparge is satisfactory for geothermal fluids up to 5,000 mg/l  $\text{CO}_2$ . If  $\text{CO}_2$  content is higher (as in some steam systems), the outlet of the first sparge bottle can be sparged through a second bottle of 2N NaOH in series.

**TABLE S.1. Summary of Samples Required for Complete Analysis of One Sample Point**

Species Determined	Sample Required	Remarks
pH	Field determination with pressurized electrodes at 25°C preferred. Rapid determination of 1 atm sample is next best.	Always record sample temperature and pressure.
Conductivity, turbidity	Raw unacidified sample (RV) in 1-liter polyethylene bottle.	1 liter is collected as back up to other samples in case bottles are lost or leak develops.
Total dissolved solids (TDS) HCO <sub>3</sub> <sup>-</sup> , CO <sub>3</sub> (by titration between pH 8.3 to pH 4.5 end points) and; SO <sub>4</sub> <sup>-2</sup> , NO <sub>3</sub> <sup>-</sup> , F <sup>1</sup> , Cl <sup>-</sup> , PO <sub>4</sub> <sup>-3</sup>	Filtered, unacidified (FU) sample in polyethylene 1-liter bottle.	
Cations (except Hg)	Filtered acidified (FA) samples. Use 500-ml polyethylene bottle containing 1. ultrapure HCl.	Analyze as soon as possible by inductively coupled arson plasma spectrometer or atomic absorption.
Hg	Use 250-ml glass bottle (FA) containing 3 ml ultrapure HNO <sub>3</sub> plus 3 ml of 5 KMnO <sub>4</sub>	
Silica (monomeric)	Careful field dilution to proper range of lab analysis, usually about 10-to-1, up to 50-to-1 dilution.	Use pipette and volumetric flask in field: dilution retards precipitation
Total carbon dioxide	Spurge gas-plus-liquid into 500 ml of 2N NaOH until 1,000 ml mark is reached.	For up to 5,000 mg/. - use two bottles in series for larger CO <sub>2</sub> content. Take blank on actual 2N NaOH used.
Hydrogen sulfide	Spurge gas-plus-liquid into 500 ml of 0.5N zinc acetate until 1,000 ml mark is reached.	Entire sample is titrated. if duplicate analyses are needed, collect two samples.
Suspended solids	Pass measured volume of liquid from cooling coil outlet through preweighed 0.45 μ filter, rinse filter with deionized water and dry.	Volume depends on solids content (100 ml to 5 liters is typical).
Gas sample	Fill 500-ml glass sample bulb with liquid from cooling coils, then invert into water to displace water out of bulb.	Shut off outlet first to slightly pressure bulb.
Dissolved O <sub>2</sub> (field only)	Pass liquid from cooling coils directly into cell for field determinations.	

TABLE S.2. Example of PNL's Bottle Labeling System

Field Location EMSA			
XX	YY	ZZ	TYPE
Two Letters Indicating System or Test	Two Numbers Indicating Sample Port	Serial No. 1 to 99	Type of Sample: RU, FU, FA, SiO <sub>2</sub> , CO <sub>2</sub> , H <sub>2</sub> S or GAS <sup>2</sup>

Example: EMSA  
BD-16-23-RU

Location East Mesa

BD: Test B Subpart D

16: Sample Port 16

23: 23rd set taken

RU: Type RAW unacidified

One Set Would Be:

BD-16-23-RU

BD-16-23-FU

BD-16-23-FA

BD-16-23-SiO<sub>2</sub>

BD-16-23-CO<sub>2</sub>

BD-16-23-H<sub>2</sub>S

BD-16-23-GAS

Weighing the bottle with 2N NaOH before and after the sampling is unnecessary if care is exercised to accurately measure the original sample volumes.

#### Measurement of H<sub>2</sub>S

The H<sub>2</sub>S sampling is similar to the CO<sub>2</sub> sampling and uses the same equipment, except that 0.5N Zinc acetate is used to stabilize the H<sub>2</sub>S for later titration. The scrubbing action of the sparge tube removes H<sub>2</sub>S from the gas.

Our tests were on systems containing only 1 to 2 mg/l H<sub>2</sub>S for which a single sparge bottle was sufficient. If the H<sub>2</sub>S content is high, a chain of two bottles in series and/or a higher zinc acetate concentration may be needed.

Colormetric field kits are very useful and convenient. Unfortunately, they only measure H<sub>2</sub>S content of liquid and not the H<sub>2</sub>S in the gas bubbles that

escape. Gas bubble loss can be minimized by keeping the coil outlet temperature below 25°C. (Some H<sub>2</sub>S will still be lost in systems containing high gas content.)

#### Measurement of Silica

Silica is usually present in geothermal waters in excess of room temperature solubility. Precipitation can be retarded by immediately diluting the sample with deionized water.

#### Sampling in Stainless Steel Cylinders

Collecting samples directly into stainless steel cylinders is not recommended, especially if the gas- and liquid-sample remains in the cylinder several days before analysis.

Several undesirable effects were observed using metal cylinders:

- Corrosion of the stainless steel can increase the measured iron and nickel by as much as 100 times.
- Flashing, which occurs when fluid enters the cylinder, can cause loss of calcium, strontium, and other elements.
- Loss of silica occurs.
- The CO<sub>2</sub> content of the gas is lowered when CO<sub>2</sub> redissolves in the liquid.
- Gas-liquid equilibrium in the cylinder after cooling causes errors in the pH measurement.
- The H<sub>2</sub>S cannot be determined accurately because no stabilization was used.

#### Measurement of Suspended Solids

The suspended solids in the liquid can be measured by passing a known volume of fluid through a preweighed filter (usually 0.45 μ). It is important to immediately rinse the filter with deionized water to wash off soluble salts that could affect the weight after drying. The test results indicate a higher suspended solids value was obtained if the sample is filtered hot, before cooling. The filter residue is very useful for x-ray and microprobe studies of the solids prepared.

A filter train arrangement of varying pore size is described in Section 9 which provides much useful solids data by particle size. A commercially available laser-beam particle counter provided very interesting data on particle size and distribution. Such data are of considerable interest to studies on the life of injection wells.

### Gas Composition

If samples of the gas are collected for composition analysis, the data should be treated with caution because the percentage levels of each gas component are strongly influenced by sampling conditions. When depressurization occurs at the coil outlet valve, gases break out of the solution much like  $\text{CO}_2$  does when a bottle of champagne is opened. The amount of  $\text{CO}_2$  released is dependent upon such variables as temperature, brine chemistry, salinity, pH, and system geometry. As more  $\text{CO}_2$  is released, the percentage composition of other components (e.g.,  $\text{CH}_4$  or  $\text{N}_2$ ) is seen to drop accordingly.

A gas composition analysis is affected by variables such as liquid temperature; we recommend that coil outlet temperature be kept at a constant  $25^\circ\text{C} \pm 0.5^\circ\text{C}$ . If all gases are soluble at  $25^\circ\text{C}$ , however a higher temperature may be required to obtain a sample.

The liquid coming out of the coils while sampling single-phase liquid systems is usually saturated with gases, so this liquid is used to completely displace the air out of the sample bulb. The bulb is then inverted into a bucket of water and the gas displaces the liquid. The bulb should be slightly pressurized with gas before closing the inlet/outlet valves.

If a steam system is being sampled, some gases will redissolve in the steam condensate, affecting both the gas percentage compositions and gas evolution rate. In some cases, most of the gas may redissolve and result in very long collection times.

### SAMPLING TWO-PHASE SYSTEMS

The sampling of two-phase systems containing  $\text{CO}_2$  liquid or combinations of steam, gas, and liquid is very difficult. So many variables come into play that it is usually impossible to use the resulting data to calculate back to an average composition of the well or pipe.

Given the state-of-the-art, we recommend that two-phase flow be directed into a full-flow, full-size separator that isolates the steam and gas from the liquid phases. The sampling methods recommended in this report can then be used to collect a complete set of data on the single-phase liquid and single-phase steam compositions. If the mass flow rates of both the liquid and steam phases are known, a "composite" analysis for the well can be calculated. If these flow rates are not known, approximate steam flash percentages can still be determined using temperature/enthalpy data to estimate the "percent steam flash", which is used to correct the data.

The main problem with sampling two-phase systems is that the "numbers" are easily obtained, but they are usually meaningless, and, therefore, lead interpreters of the data into drawing erroneous conclusions. Such data are worse than useless. Some of the problems of sampling two-phase systems are as follows:

- The flow in the pipe is unstable. Conditions are rapidly changing at the probe inlet, requiring the use of large numbers of samples to get reasonable statistics for one point.
- The flow in the pipe is nonuniform, and hence, arriving at an average flow profile is difficult. Stratification is often a problem, requiring probe traverses in vertical, horizontal and diagonal directions. If the system flow is characterized by slugs or oscillations of liquid such probe traverses produce unreliable data.
- The probe may not pick up the proper ratio of steam and gas-to-liquid. Depending only on probe orientation and sample flows, we observed variations of gas-liquid ratios of over a factor of 100.
- Isokinetic sample probes are required for sample measurements in two-phase systems. The isokinetic probe includes pressure taps to assure better pick-up of the correct steam-to-liquid ratio. However, such probes are not yet developed and are expensive to build and operate. And even if they work perfectly, a mini-research project is required to get all the data to arrive at a statistically valid sample of the average pipe chemistry.

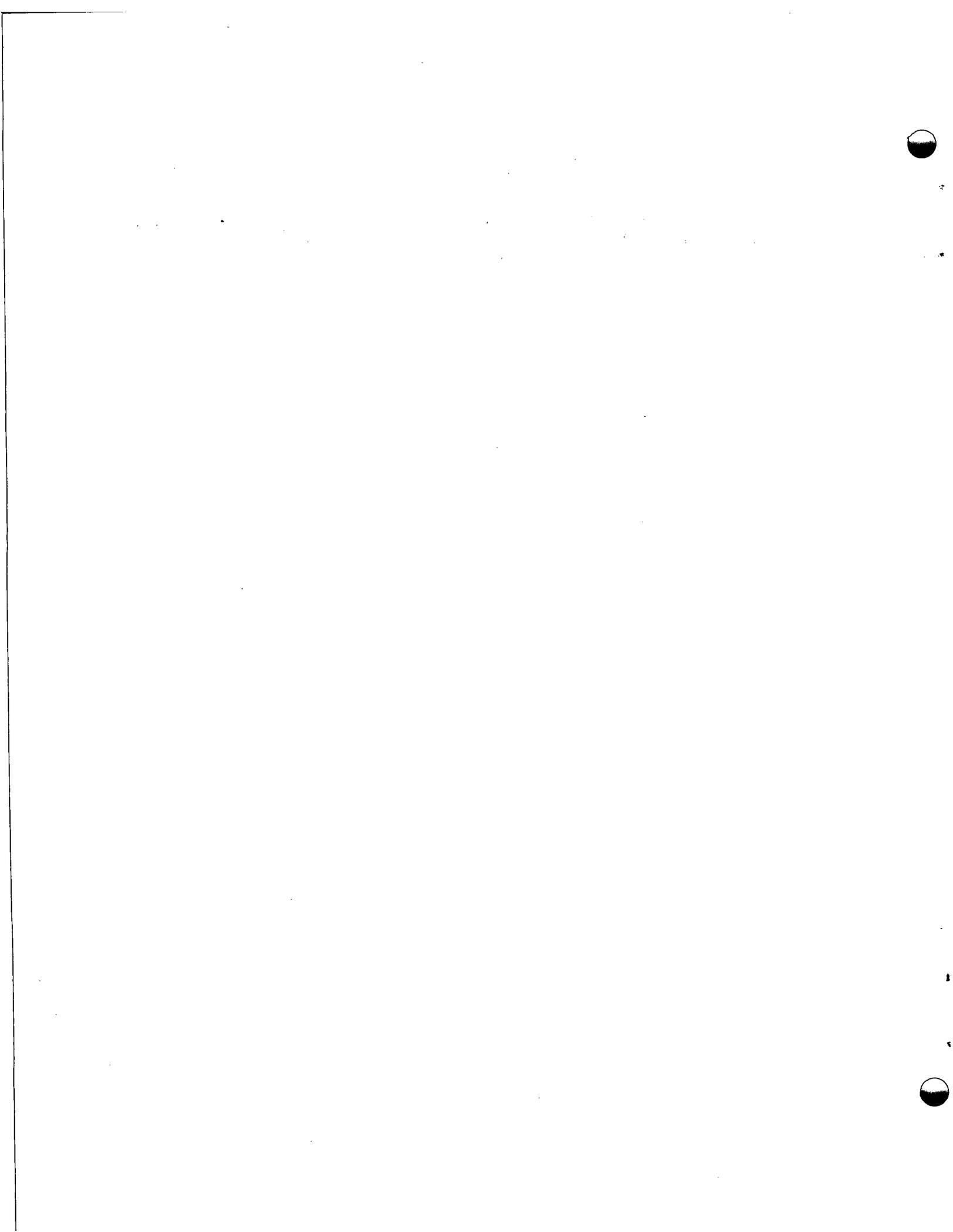
Small Webre separators were used on the outlet of a sample probe in order to separate the steam and gas from the liquid. We were able to determine the liquid content and composition, steam content and composition, and gas content and composition at every point, but our measurements are subject to error due to our not sampling with an isokinetic probe. Our most reliable data were obtained with a 1/2" diameter, non isokinetic, sample probe, coupled to a Webre separator with 1/2" tubing and a ball valve, operated to produce almost no pressure drop between the loop and separator. The steam and liquid were then sampled using two sets of double-sampling coils. Had an isokinetic probe been used, some interesting traverses of the pipe could have been made.

Further development of two-phase sampling methods is suggested along the following lines:

- Develop in-line mixers to homogenize the two-phase flow, so that the sample probe has a chance to pick up correct, average steam:liquid ratios for the pipe as a whole.
- Develop an isokinetic probe to sample the mixer so that correct steam:liquid ratios are extracted from the pipe.
- Complete development of small analytical separators for determining the steam, liquid, and gas ratio, and for collecting samples of each for analysis.
- Run field tests. Prepare detailed procedures for such sample systems.

## REFERENCES

1. T. S. Presser and I. Barnes. "Special Techniques for Determining Chemical Properties of Geothermal Water." U.S. Geological Survey Water Resources Investigations, 22-74, August 1974.



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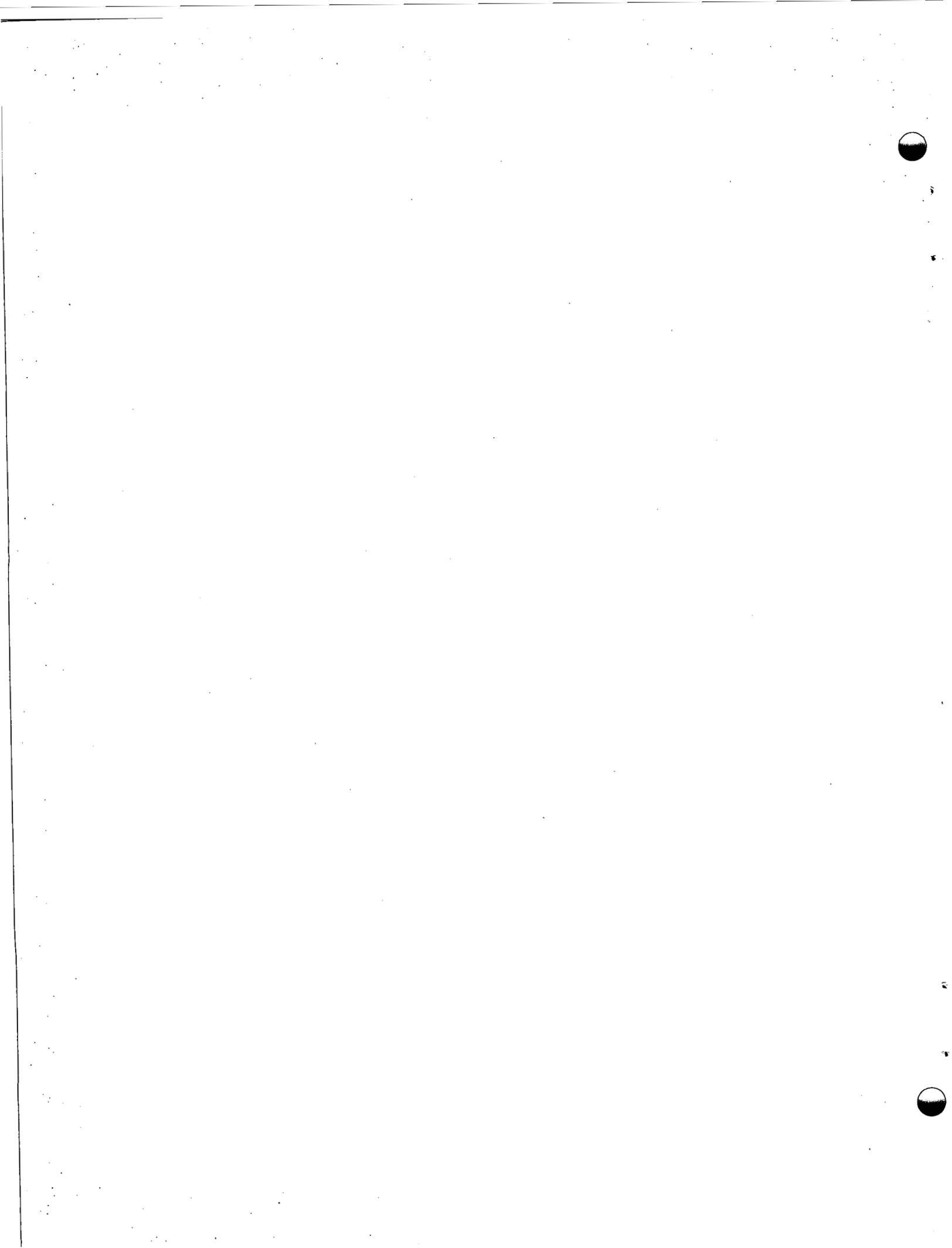
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## INTRODUCTION

In 1978, Pacific Northwest Laboratory published a manual of geothermal sampling and analysis methods.<sup>(1)</sup> This manual collected and summarized most of the methods then in use for taking and analyzing geothermal samples. No attempt was made to identify the best methods available or to standardize methods. The reader had to decide for himself for a particular application.

The objective of the work presented in this report was to test and compare many of the sampling methods cited in the manual under a single set of controlled field conditions, leading to recommendations on how samples should be taken and preserved. The next step will be to standardize sampling procedures for single-phase liquid and steam systems. Sampling of a two-phase system presents considerable difficulties which we discussed in this report. We do not believe that two-phase sampling procedures can be standardized at this time.

This field test was conducted over a two week period in August 1979 at the U.S. Department of Energy Geothermal Component Test Facility at East Mesa, California. We received the complete cooperation of DOE and the East Mesa Site staff.

The geothermal well was flowed in an artesian flow mode to minimize flashing. Although some gas breakout did occur, almost no steam flash occurred between the well and the sampling test loop. We collected triplicate samples at the wellhead at the beginning of the test and at the end of the two week test period. The test-loop inlet (which was almost single-phase) was sampled daily with triplicate samples for each analytical determination. These samples of the single phase fluid represent the closest values to the true composition of the fluids.

After entering the loop, the geothermal brine was then passed through a flow orifice in order to drop the pressure just enough to cause significant CO<sub>2</sub> gas breakout and permit sampling of two-phase CO<sub>2</sub>-brine mixtures. The brine there was then passed through a second orifice, where a steam flash occurred, and two-phase brine-steam samples were taken.

We evaluated procedures for taking liquid, gas and suspended solid samples. Each method is described in detail in the body of this report.

## REFERENCES

1. J. C. Watson. "Sampling and Analysis Methods for Geothermal Fluids and Gases." PNL-MA-572.

## 1.0 SAMPLING TEST LOOP AND TEST PAD ARRANGEMENT

E. M. Woodruff

### 1.1 TEST SITE

The sampling tests were conducted at the Geothermal Component Test Facility (GCTF) at East Mesa, California. This complex of supply and injection wells, test area and supporting shops and laboratories is operated for the Department of Energy by Westec Services, Inc., to provide geothermal fluid and services to experimenters interested in studies related to geothermal energy extraction. Arrangements prior to testing included formal application to DOE's San Francisco Operations Office and execution of a test agreement.

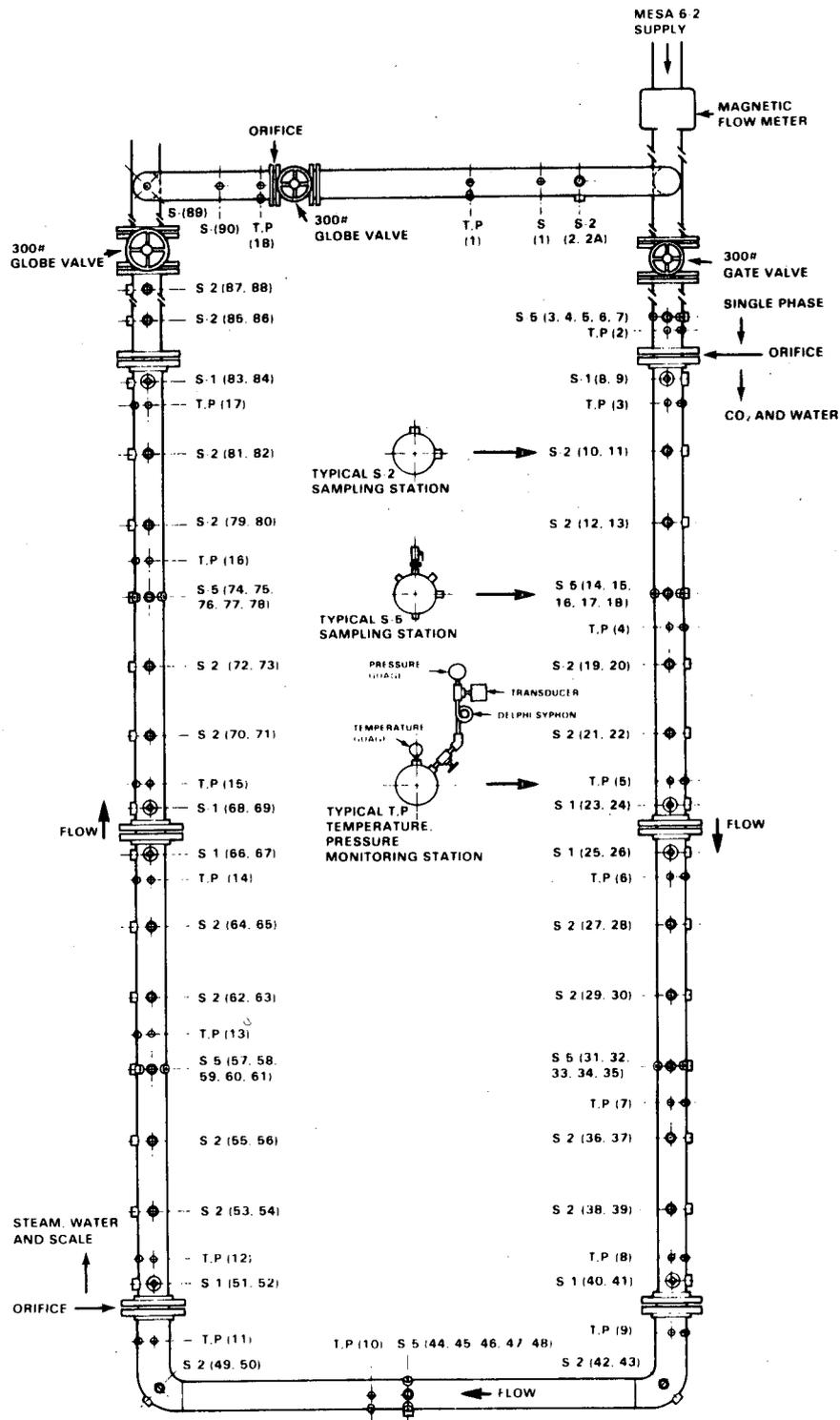
The operator reserved Area #3 on the test pad and scheduled flow from Mesa Well 6-2 with the stipulation that flow during the test period be maintained as constant as possible to minimize brine chemistry perturbation.

### 1.2 TEST LOOP

A test loop was designed by PNL<sup>(1)</sup> based on a concept<sup>(2)</sup> developed and operated by Republic Geothermal, Inc., at their well field north of the GCTF. In principle, the loop permits diversion of all or part of the flow from a well through a series of pipe spools that incorporates access ports with sampling or monitoring capability.

Spool junctions permit insertion of orifice plates with openings designed to provide a series of pressure reduction stages to simulate flow conditions of interest. A bypass leg and inlet and outlet valves complete the essential loop components.

Figure 1.1 illustrates the loop designed for these tests. Access ports and their numeric identification are shown. Several types of sampling stations are illustrated in the center of the figure. These provide penetrations on the top and side in type S-2 Stations for vertical or horizontal traversing or multiple access at the same location. S-5 Stations add ports at positions



**FIGURE 1.1.** Test Loop Schematic, Numbered Ports Are Located Around the Perimeter, Station Details at the Center

45° from vertical and horizontal. Another type of station not illustrated (S-1) provides a sampling port on top of the loop and a larger opening on the side for monitoring probe devices.

Temperature and pressure monitoring stations, T-1 through T-18 and P-1 through 18, respectively, provided temperature and pressure gauges for visual observation typically at the inlet, center, and outlet of each pool. Sensors also coupled these locations to a data logging system for recording both temperature and pressure.

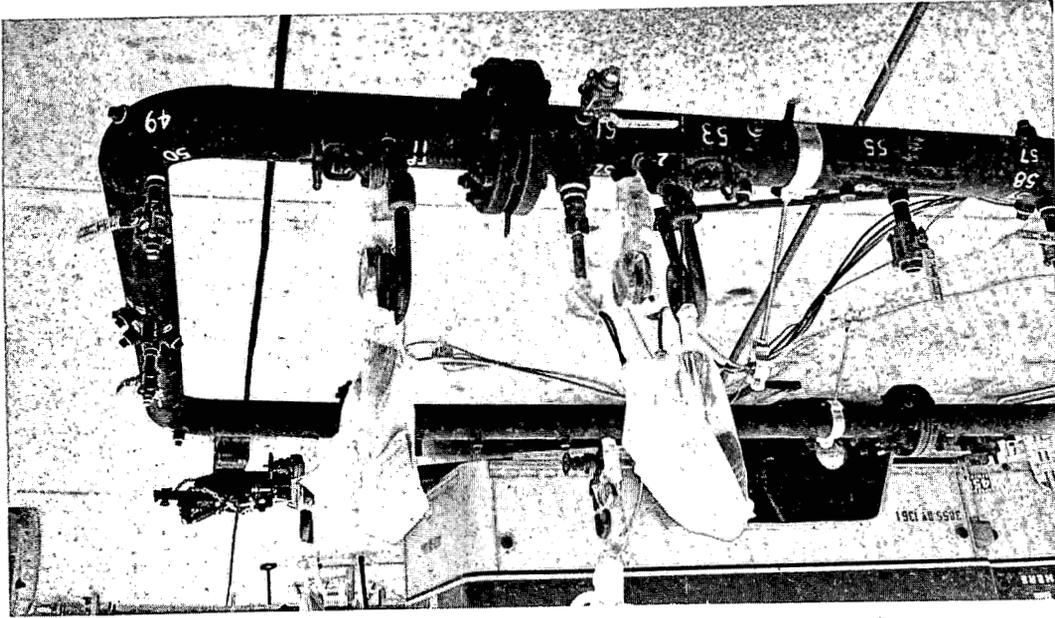
A local fabricator was contracted to build the loop, install it on the test pad, and after completion of tests, disassemble and transport the components to storage. Ball valves were installed on ports scheduled for repeated use and spare openings were plugged. Figure 1.2a and b show the loop after installation on the test pad. The arrow on the pipe in the lower right of Figure 1.2a shows the direction of fluid flow. The spool connection just left of T- and P-11 in Figure 1.2b contains the second orifice plate as evidenced by a short handle protruding from the top. The pipe is 4" schedule 40 ASTM A-53 Grade B.

Initial sizing of orifices provided a 1.1" diameter inlet opening and a 1.0" diameter second orifice. During the second week of sampling, this second orifice was changed to 0.8" diameter. This design produced three sampling regimes: single-phase fluid at the inlet, two-phase gas and liquid after the first orifice, and three-phase steam and gas, liquid, and scale downstream from the second orifice.

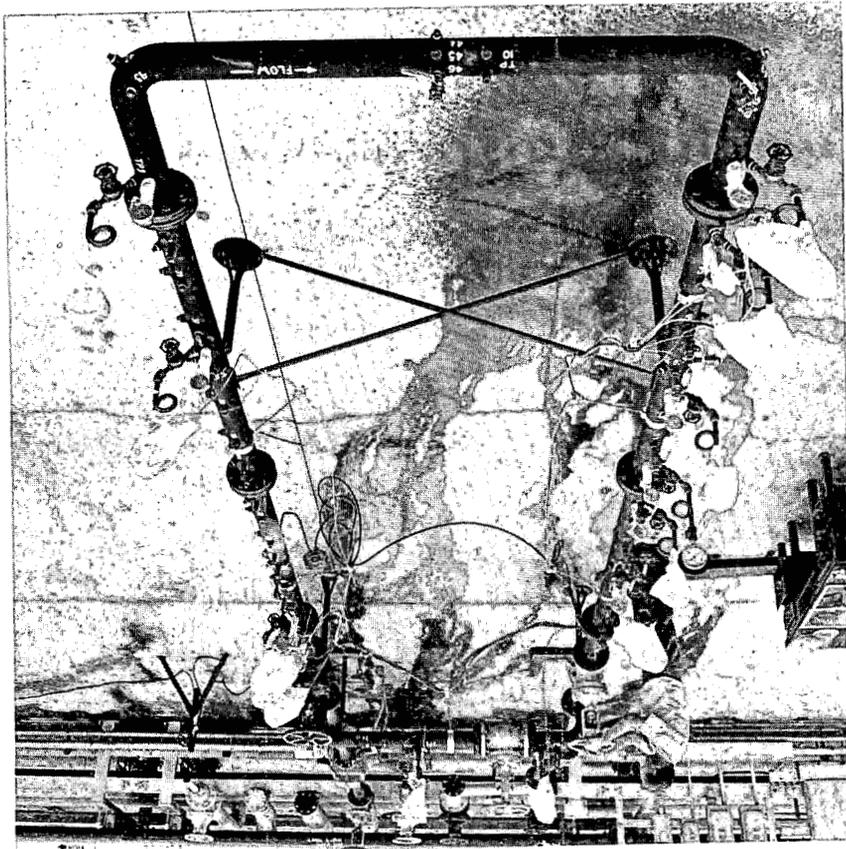
### 1.3 SUPPORT FACILITIES

To complete the installation, an air-conditioned office trailer was positioned adjacent to the loop to house data logging capabilities and instruments including a laser particle counter and a laboratory pH meter. Office space provided on-site was also utilized for weighing, component assembly, and phone access. The trailer used in towing supplies to the site was parked on the opposite side of the loop and housed test supplies in numbered boxes arranged

FIGURE 1.2. Sampling Test Loop: (a) Overview Looking Toward Supply and Return Manifolds; (b) Ports Near the Second Orifice Left of T-and P-11



(b)



(a)

in the sequence of use. A tarpaulin was rigged over the loop to provide shade for the experimenters. (This luxury shortly succumbed to another desert pleasantry -- a thundershower replete with 1" diameter hailstones.)

## 1.0 REFERENCES

1. S-2 Test Loop Plan, Section and Details. Drawing No. SK-3-21422, Pacific Northwest Laboratories, Battelle Memorial Institute, Richland, WA, July 2, 1979.
2. O. J. Vetter, D. A. Cambell and M. J. Walker. Geothermal Fluid Investigations at RGI's East Mesa Test Site. PNL-2556.

## 2.0 SAMPLING LOOP OPERATION

E. M. Woodruff and O. H. Koski

### 2.1 LOOP START-UP, SHUT DOWN

Starting the loop for a day's operation involved diverting the flow from the bypass leg (see Figure 1.1) into the loop by opening the outlet globe valve and the inlet gate valve simultaneously and then closing the globe valve in the bypass line so that the loop received full flow. The reverse of this procedure was used to shut the loop down and transfer flow to the bypass leg at night. This procedure maintained continuous well flow 24 hours a day, thus avoiding fluctuations in brine chemistry associated with valving flow at the wellhead.

### 2.2 DATA LOGGER AND PROCESS INSTRUMENTATION

Flow conditions in the loop were manually monitored by taking gauge readings at intervals of one to two hours during sampling operations and by automatically scanning the system at five minute intervals with a data logger coupled to magnetic tape storage. Figure 2.1 summarizes the sensor, processing, and storage elements assembled in the data logging system.

### 2.3 OPERATING SCHEDULE

The sequence of events involved in loop operation and testing is summarized in Table 2.1. Delivery of components prior to start-up began on August 3, 1979. Assembly and pressure testing was complete by August 9, and monitoring sensors and the data logger were operational on August 13. Testing supplies arrived by trailer on August 10. The test schedule as shown in Table 2.1 includes a daily sampling of the loop inlet brine chemistry (Section 6) and complete analyses of wellhead chemistry at the beginning and end of loop operation. Major perturbation of the loop conditions resulted from scale build-up in the flashing section downstream of the second orifice. Corrective acid treatments are listed in the table. Injection of HCl had the immediate effect of dissolving the calcium carbonate scale and restoring normal flow and pressure conditions.

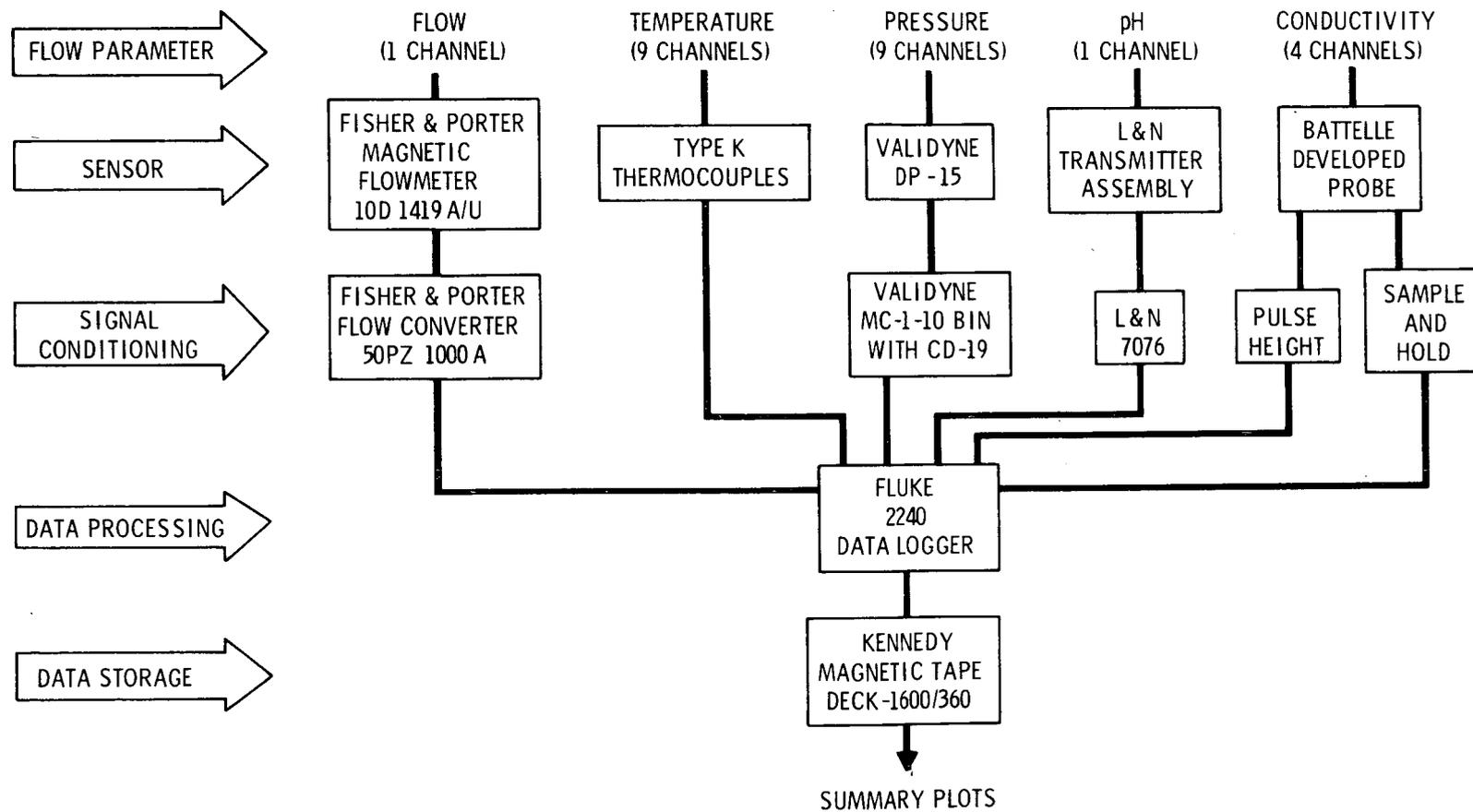


FIGURE 2.1. Data Logging System Sources, Signal Flow and Processing Equipment For Data Logging System

TABLE 2.1. Schedule of Loop Operation and Tests

Event	Calendar (August)/Julian Days														
	14/ 226	15/ 227	16/ 228	17/ 229	18/ 230	19/ 231	20/ 232	21/ 233	22/ 234	23/ 235	24/ 236	25/ 237	26/ 238	27/ 239	28/ 240
Check and Calibrate	x														
Test 1, Sample Wellhead		x													x
Test 2, Sample Loop Inlet		x	x	x	x	x	x	x	x	x	x	x		x	x
Test 3, pH Measurement				x											
Test 4, CO <sub>2</sub> Absorption			x												
Test 5, H <sub>2</sub> S Sampling			x	x					x						
Test 6, Evacuacted Cylinders								x							
Test 7, Suspended Solids				x	x										
Test 8, Non-Condensibile Gas							x	x							
Test 9, Separators/2 phase supply									x	x	x				
Test 10, Separators/1 phase supply										x	x				
Test 11, Isokinetic Probe								x							
Acid Treat Scale					x		x		x	x		x		x	
Change Second Orifice 1.0 to 0.8							x								
Thunder Storm			x												

2.3

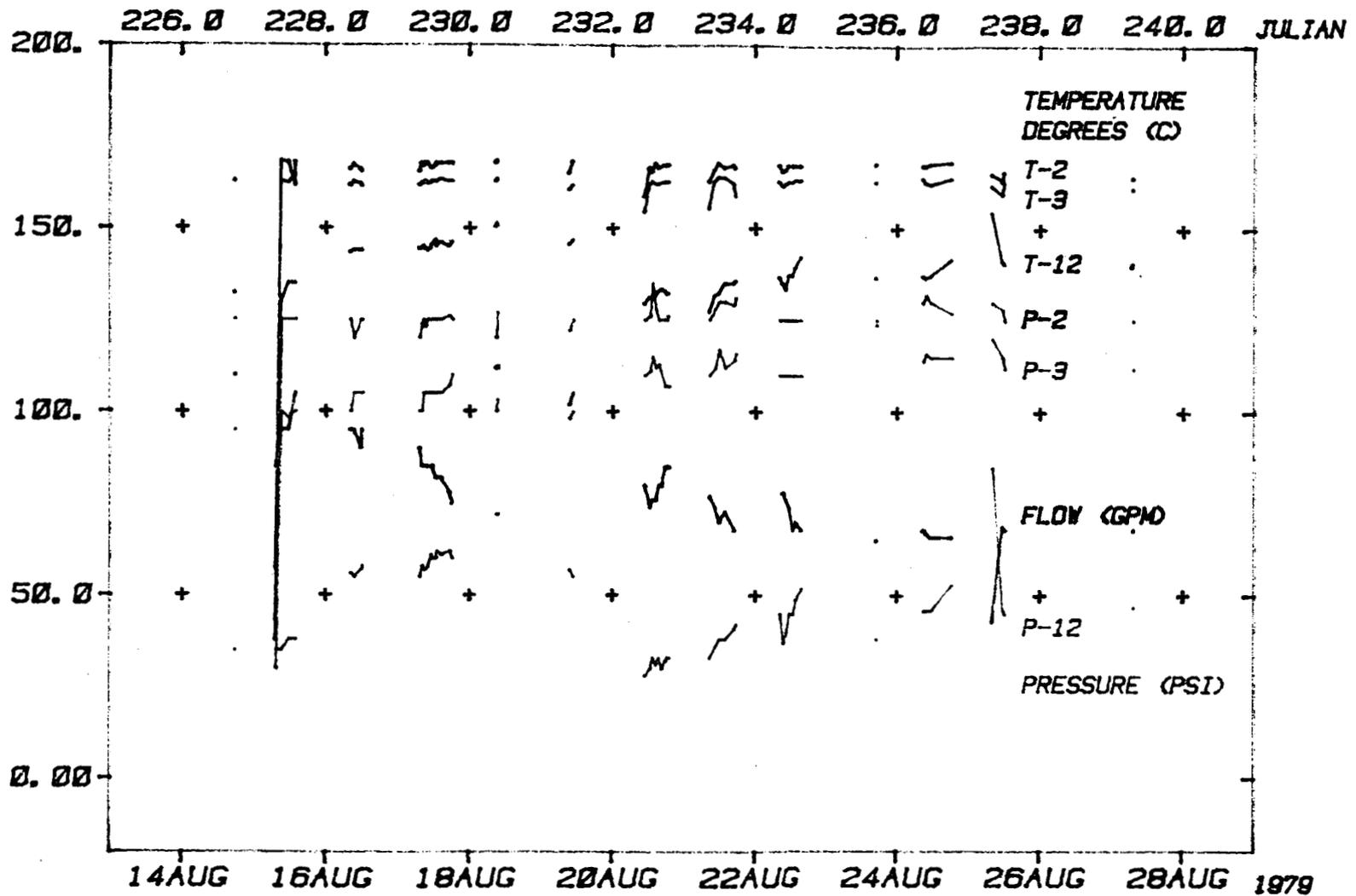


FIGURE 2.2. Manually Recorded Pressure, Temperature and Flow Inlet Conditions and After First and Second Orifices As Located in Figure 1.2

## 2.4 FLOW DATA

Plots of manually recorded data are given in Figure 2.2. Information retrieved from magnetic tape that summarizes a typical day's operation appears in Figure 2.3. Figure 2.4 extends that analysis over a period of several days. We further processed manually logged data by entering them on tape for storage, retrieval, and plotting in the same format as information collected by the data logger system. The data points selected illustrate changes in conditions as fluid progresses through the test loop. Thus, in each data logging scheme, Plots T-2 and P-2 are the inlet temperature and pressure, respectively, T-3 and P-3 are the temperature and pressure after passage through the first orifice, and T-12 and P-12 are in the steam flash regime produced by the second orifice.

These numbers also correspond to the respective temperature and pressure (T, P) monitoring stations located on the loop in Figure 1.1.

In Figure 2.3, scale rate and conductivity are included for sensors located in Sample Port S-18. This section of the loop containing the water-plus-CO<sub>2</sub> regime produces slight scaling effects, making it possible to observe probe performance without rapid fouling of the probe that would occur under steam flash conditions. Response of the scale rate probe on August 18th (Figure 2.3) suggests sensitivity to surface film changes following the acid treatment applied to the loop prior to start-up that day. Interpretation of probe output in terms of film or scale properties would require further calibration. Note that at about 6:30 p.m., both scale rate and conductivity readings go off scale. This resulted from a manual adjustment of gain.

Calibration of the conductivity probe output prior to its installation in the loop established a relationship of 200 mv/mmho. Conductivity for the period shown is on the order of 1.5 mmhos.

## 2.5 GEOTHERMAL TEST FACILITY RECORDS

Records kept by Westec, Inc., for Well 6-2 are summarized in Table 2.2. Readings from several days prior to start-up of the loop are included as confirmation that well flow was stabilized before the test series began.

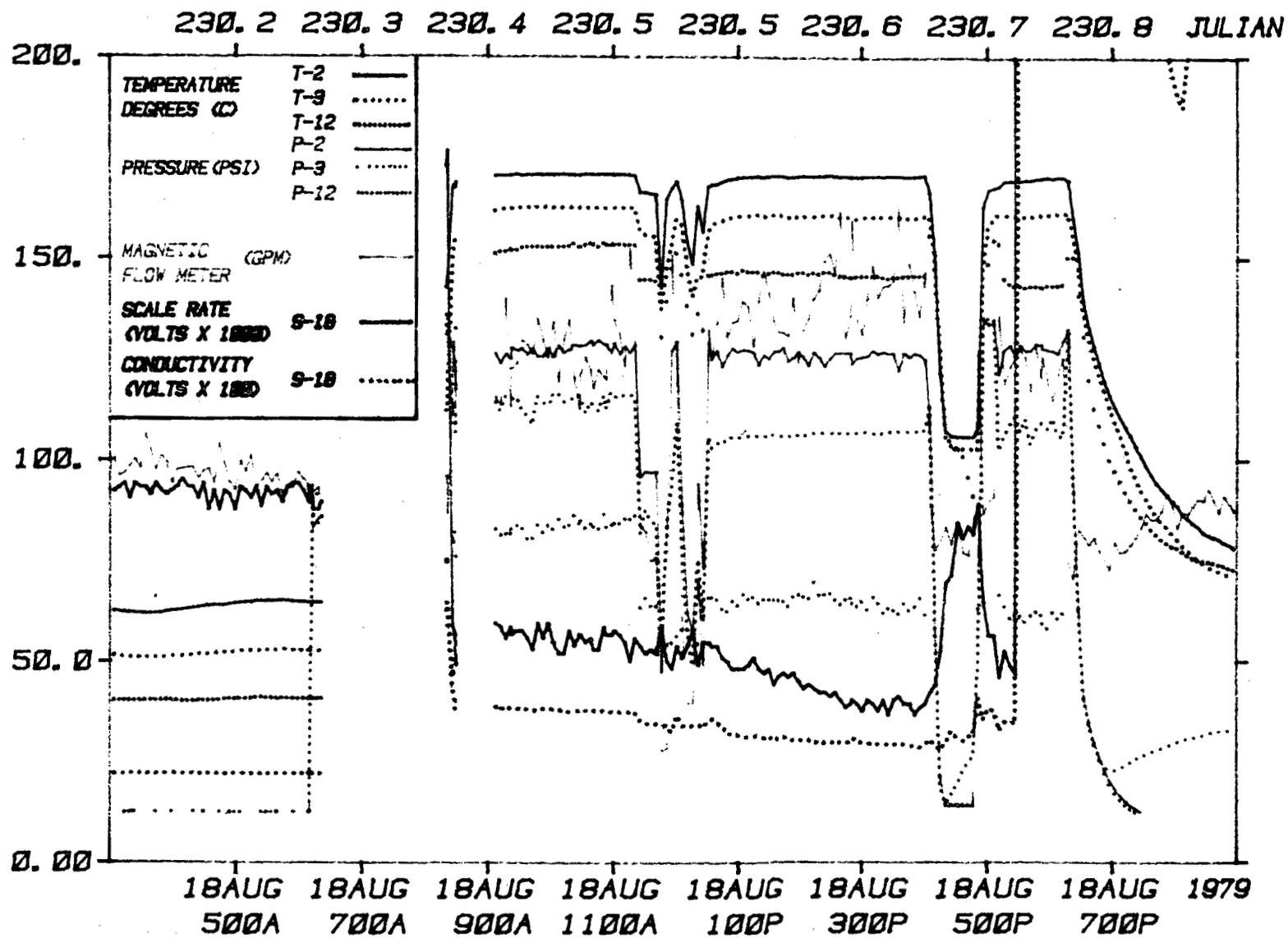


FIGURE 2.3. Data Logger Record - Pressure, Temperature, Flow, Conductivity and Scaling; Expanded for One Day (August 18, 1979)

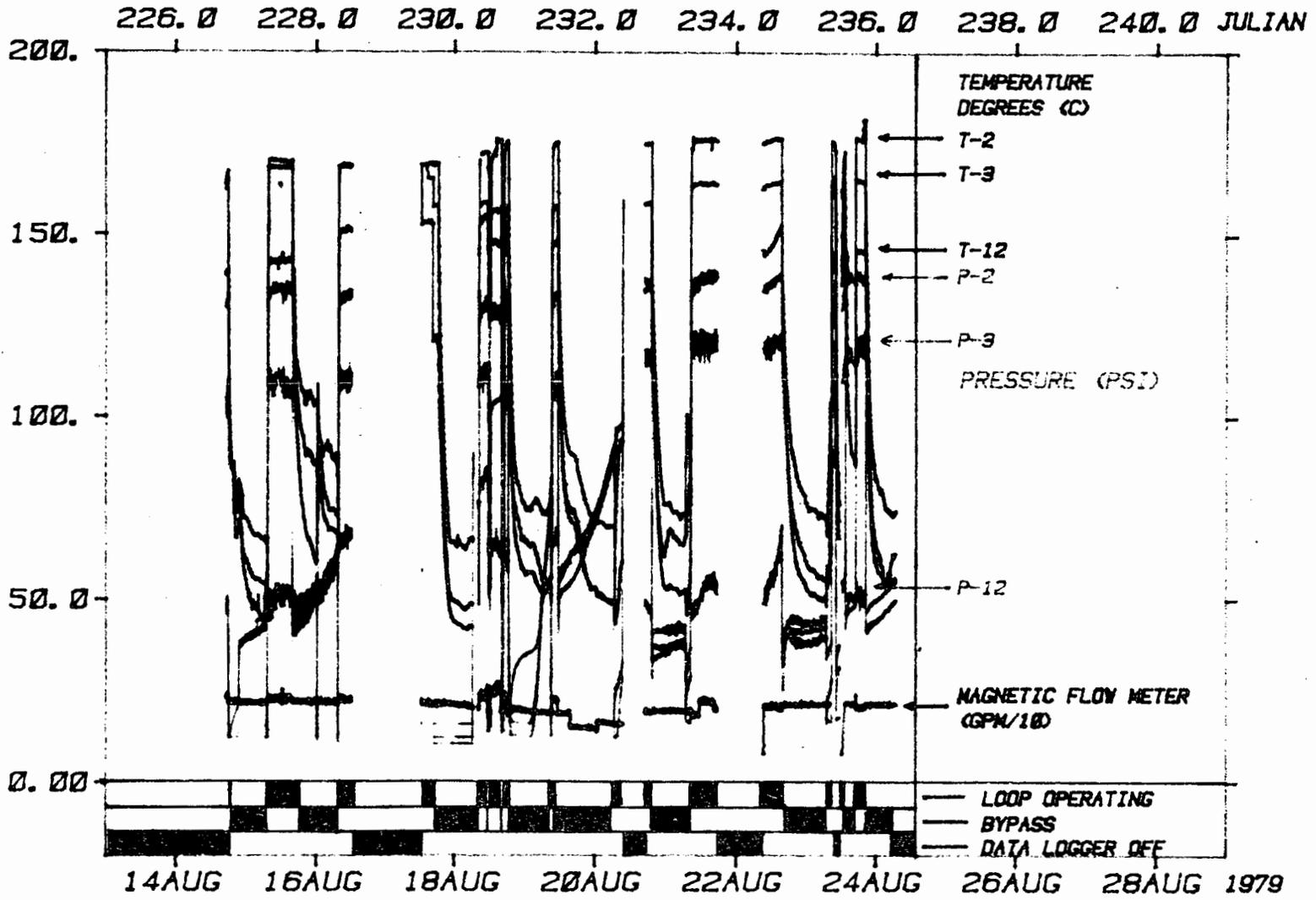


FIGURE 2.4. Data Logger Record - Summary of Test Period, Temperature, Pressure, and Flow

TABLE 2.2. Geothermal Well Mesa 6-2 Status  
(From Westec, Inc., Wellhead  
Logs and Charts)

<u>Date Calendar/Julian</u>	<u>Pressure Psig<sup>(a)</sup> (Taken at 9:00 a.m.)</u>	<u>Temperature Ave. °F<sup>(b)</sup></u>	<u>Flow Ave. gpm</u>
August 12/224	160	330	100
August 13/225	160	320	100
August 14/226	150	320	100
August 15/227	160	340	100
August 16/228	150	340	100
August 17/229	149	340	100
August 18/230	149	340	100
August 19/231	150	340	100
August 20/232	150	340	100
August 21/233	149	340	100
August 22/234	149	340	100
August 23/235	140	340	100
August 24/236	152	340	100
August 25/237	154	340	100
August 26/238	152	340	100
August 27/239	152	340	100
August 28/240	148	340	100

(a) Pressure from wellhead guage.

(b) Temperature at a point ~20' downstream from wellhead.

Flow data indicate a more stable condition than suggested by data from the flow meter on the test loop. Well data represent an average daily estimate; variations in the loop flow data represent responses to scaling and changes in the flow pattern (bypass versus loop) that are independent of total flow. They are independent because the loop was connected parallel to the supply manifold on the test pad.)

### 3.0 WELLHEAD SAMPLING (TEST 1)

E. M. Woodruff

#### 3.1 INTRODUCTION

This is the first in a series of chapters that describes and evaluates test procedures. Tests 1 and 2 are identical in their method for sampling single phase systems and were applied simultaneously at the wellhead and the test-loop inlet. These tests provided a basis for fluid chemistry control for all later tests. Test 2 developed fluid characteristics on a daily basis so that other tests spanning more than one day could factor in fluid stability. Methods employed in Tests 1 and 2 were chosen to provide a baseline because of their proven effectiveness over a four-year program. Sampling methodology is described for the benefit of other users and for comparison with other tests in which more emphasis is placed on methodology.

#### 3.2 OBJECTIVE

Test 1 was designed to establish wellhead brine chemistry (as near to single-phase as possible) at the beginning and end of the test period. It served with Test 2 as a control for all other tests. The test also helped us to determine whether or not deposits collect on the internal surfaces of cooling coils, thus lowering the apparent concentration of certain elements.

#### 3.3 SAMPLING POINT

Wellhead samples actually came from two locations shown in Figure 3.1. When the total CO<sub>2</sub> contents of the wellhead samples was compared with samples from the loop, higher values were initially found at the wellhead. The sample point was then moved to avoid gas enhancement by sampling pipe configuration, which was uncertain at the time.

#### 3.4 SAMPLING METHOD

Wellhead sampling was performed with a system referred to as the double coil method (see Figure 3.2). Fluid passes through two 1/4" stainless steel

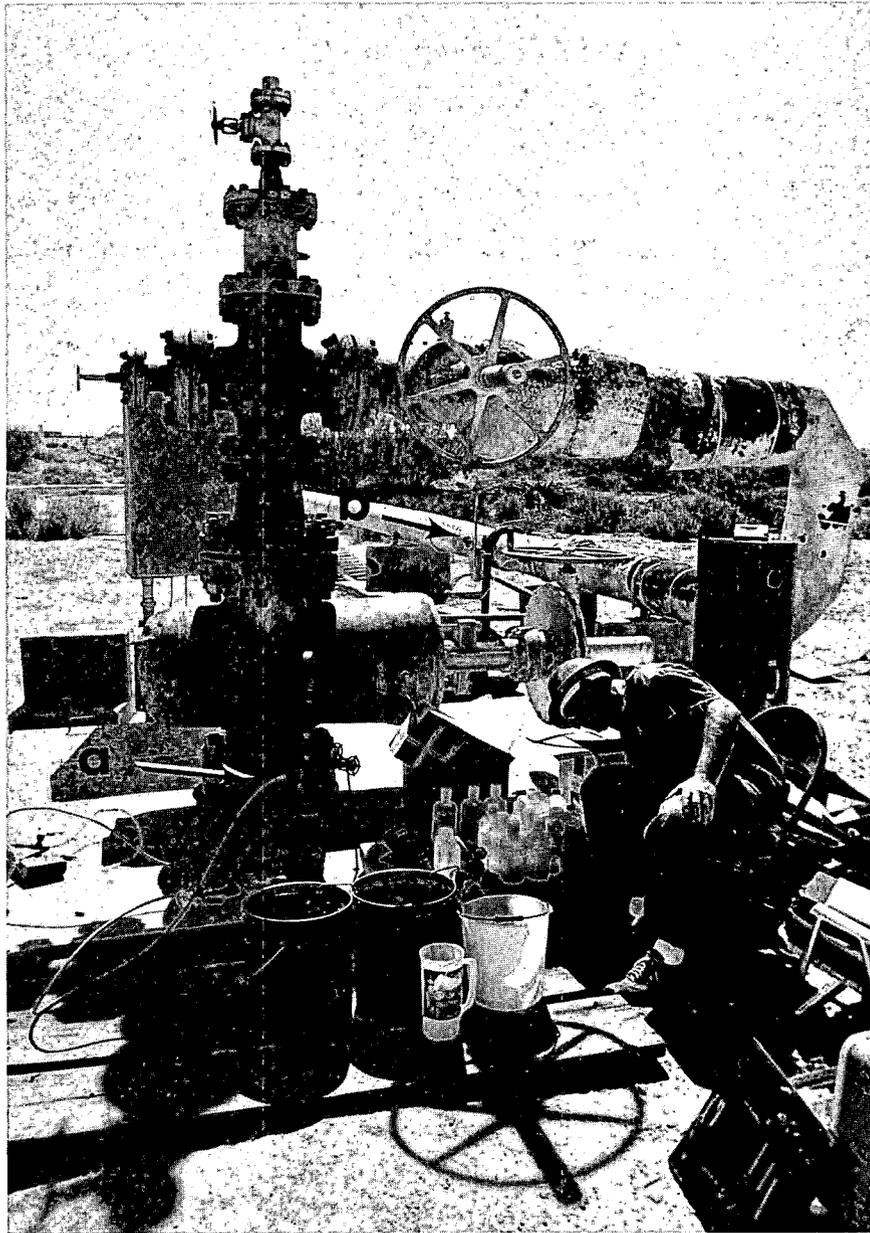


FIGURE 3.1. Mesa 6-2 Wellhead Sample Points: Arrow (a) Locates First Sample Point; Arrow (b) Locates Second Sample Point Used at End of Tests

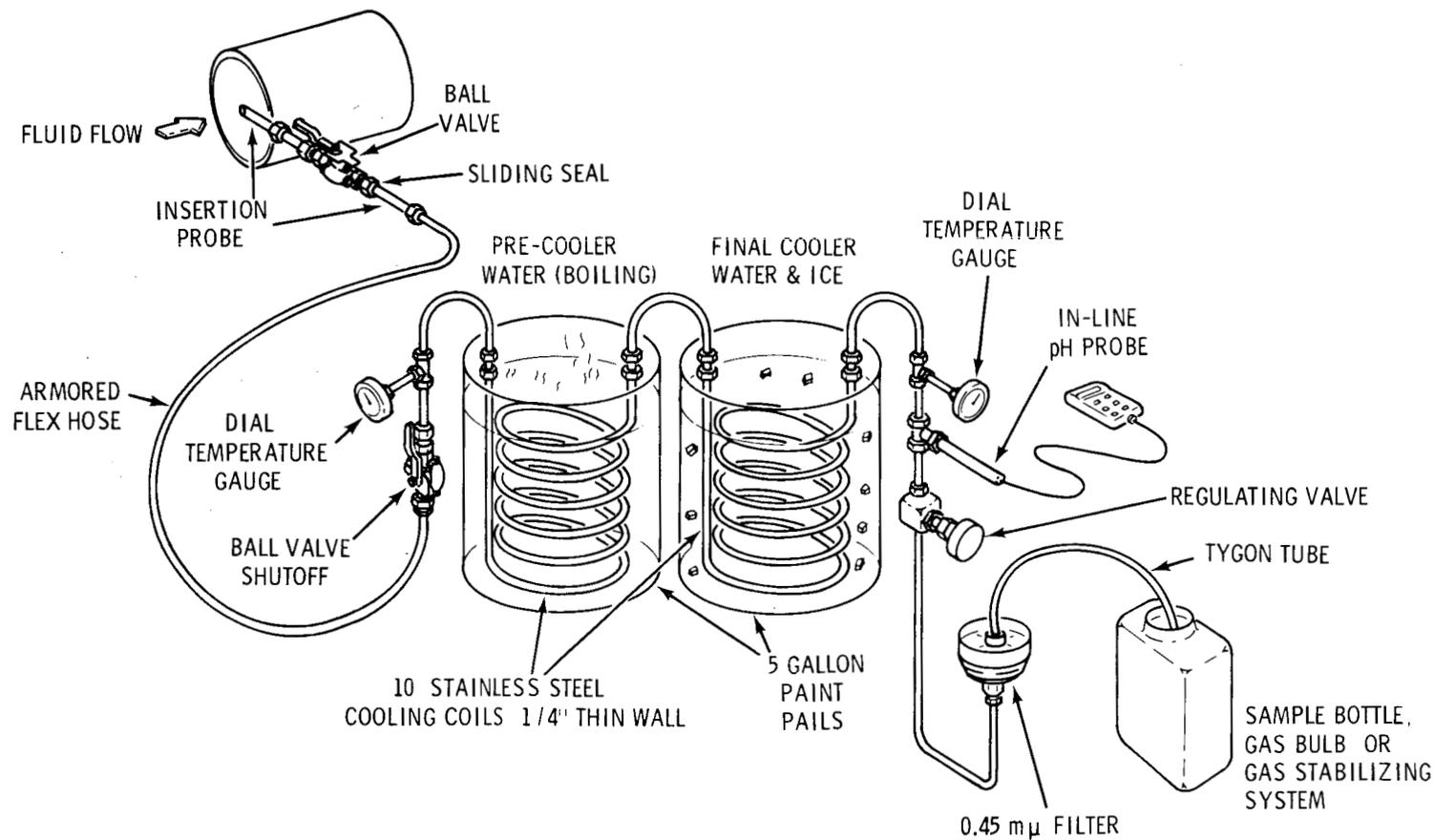


FIGURE 3.2. Test 1 Sampling Assembly

cooling coils, the first placed in a bucket filled with water, and the second in a bucket filled with ice water. Flow is regulated downstream of the coils to prevent steam flashing before cooling and to deliver a cool sample stream that can be treated in a number of ways in order to satisfy different analysis. Triplicate samples were collected for each determination. A complete list of equipment and sampling procedures is included in Section 13. An in-line pH cell should be used only if it can withstand full system pressure. A complete procedure for this method is given in Section 13.

### 3.5 RESULTS

Table 3.1 lists analytical results for the initial and final days of operation. Averages and standard deviations are given for samples taken in triplicate. Data for samples taken simultaneously at the test loop inlet (Test 2) are included for comparison. A general trend of slight reductions in concentrations over the test period is indicated. With the exception of the CO<sub>2</sub> comparison on the initial day, test loop inlet and wellhead results are in general agreement. Gas breakout was insufficient at the sample point to collect a gas sample for mass spectrometer analysis.

TABLE 3.1. Analytic Results for Test 1 at Wellhead and Test 2 at Loop Inlet

<u>Parameter/Sample ID</u>	<u>A-00-01, 02, and 03 Wellhead On 8/15/79</u>	<u>B-03-01, 02, and 03 Loop Inlet On 8/15/79</u>	<u>B-03-47, 48, and 49 Loop Inlet On 8/28/79</u>	<u>A-0A-07, 08, and 09 Wellhead On 8/28/79</u>
pH Field	5.9 @ 37°C	6.12 ± 6	5.95 ± .02	5.9 @ 33°C
Conductivity	6520 ± 139	6640 ± 69	6308 ± 273	5976 ± 157
TDS	4640 ± 294	4248 ± 7	4245 ± 5	4254 ± 10
Suspended Solids				0.42
HCO <sub>3</sub> <sup>-</sup> (Titration)	617.9 ± .2	595 ± 14	556.7 ± 12.5	578.6 ± 1.8
SO <sub>4</sub> <sup>-</sup> (Ion Chrom.)	128.4 ± .6	161 ± 2	158.9 ± 0	160.7 ± 0
F <sup>-</sup> (Ion Chrom.)	2.9 ± .06	3.0 ± .1	3.6 ± .3	3.4 ± .06
Cl <sup>-</sup> (Titration)	1931 ± 19	1958 ± 7	1940 ± 3	1922 ± 17
NH <sub>3</sub> (Electrode)	13.8 ± .2	14.8 ± .4	15.3 ± .12	--
SiO <sub>2</sub> (Colorimetric)	242 ± 0	246 ± 6	205 ± 6	22- ± .20
<u>OTHER</u>				
Total CO <sub>2</sub>	1256 ± 41	1068 ± 62	1320 ± 38	1319 ± 41
Field H <sub>2</sub> S	0.9	0.8	0.7	0.6
Field NH <sub>3</sub>	12	12	--	20
O <sub>2</sub> (ppb)	5	5	<5	<5
<u>Inductively Coupled Argon Plasma</u>				
Al				
As	0.58 ± .03	.5 ± .2	.55 ± .20	0.6 ± .06
B	7.2 ± .2	7.5 ± .1	7.6 ± .1	7.2 ± .3
Ba	.3 ± 0	.3 ± 0	.3 ± 0	.3 ± 0
Ca	10.7 ± .2	8.2 ± .2	10.7 ± .1	10.1 ± .6
Fe	.48 ± .03	0.2 ± 0	0.07 ± .02	0.16 ± .05
K	122 ± 1	113 ± 6	120 ± 0	123 ± 2
Li	4.65 ± .1	5.3 ± .1	5.5 ± .5	4.9 ± .4
Mg	.11 ± .02	.3 ± 0	0.4 ± .06	.10 ± .04
Na	1456 ± 22	1578 ± 12	1595 ± 79	1500 ± 61
P	0.1	--	--	0.1
Si	100.7 ± 1.8	102.2 ± 1.7	108.8 ± 2.7	101.8 ± 2.8
Sr	2.2 ± .06	2.2 ± 0	2.2 ± .06	2.3 ± .1

## 4.0 TEST LOOP INLET SAMPLING METHODS AND RESULTS (TEST 2)

D. D. DeMonia and M. W. Cole

### 4.1 INTRODUCTION

Each day a complete analysis of the test loop inlet was made to obtain a control set of data in order to establish a reference for all other tests run on the loop. Other objectives were to determine brine composition variances, if any, between the wellhead and the test loop inlet and to detect daily variations in brine composition.

### 4.2 SAMPLING METHODS

Samples were collected from Port 3 located in the single-phase section of the test loop (see Figure 1.1). All samples were taken at Port 3, the horizontal position, except the gas samples which were extracted from Port 5, the vertical position, where a small gas bubble formed upstream of the orifice. A 1/4-inch sample probe was inserted into the center of the pipe with the inlet of the probe directed into the brine flow. Samples were collected from a cooling coil assembly (see Figure 3.2). Triplicate samples were collected each day after sufficient time was allowed for the test loop to become stabilized.

### 4.3 SAMPLES COLLECTED

Raw brine (labeled RU) was collected for pH, conductivity, alkalinity ( $\text{CO}_3^-$  and  $\text{HCO}_3^-$ ), and silica. Silica samples were diluted 1:50 at the time of sampling. Filtered brine was collected through a  $0.45 \mu$  filter for laboratory determination of total dissolved solids and anions. Filtered, acidified (1% HCl) samples (labeled FA) were taken for cation analyses (acid stabilization retards precipitation); filtered, unacidified brine (labeled FU) samples were taken for anion analyses. Five liters of the brine was passed through the in-line  $0.45 \mu$  filter for suspended solids determinations. Five hundred milliliters of brine was sparged into 500 ml of 2N sodium hydroxide solution for total  $\text{CO}_2$  analyses. Conductivity and pH measurements were made at the beginning of each sampling period. Analyses for hydrogen sulfide, oxygen, and

ammonia were also done during each sampling period using commercially available field testing kits. Gas samples were taken using procedures discussed in Section 10.

#### 4.4 DISCUSSION OF RESULTS

Table 4.1 illustrates the typical brine composition during the testing period based on triplicate samples taken on each day for 13 days. Most results except for pH, alkalinity ( $\text{HCO}_3^-$ ), total carbon dioxide and calcium varied less than 5%. (The iron, arsenic and magnesium values also varied more than 5%. However, all of these concentrations were near the detection limits for the analytical method utilized. Ammonia values had a relative standard deviation of 9.7%).

During the two-week operation of the test loop, calcium carbonate scale was formed downstream at the second orifice in the loop. This caused the brine flow to decrease. On six separate occasions during the testing period, it was necessary to acidize the test loop to make it operational (Table 4-2).

Total carbon dioxide values in the inlet brine increased when the loop flow decreased. The decreased flow rate caused an increased pressure at the test inlet that kept more  $\text{CO}_2$  in solution (Figure 4.1 and Table 4.2).

The calcium concentration increases with increased  $\text{CO}_2$ . The higher calcium content of the brine is probably a result of the higher  $\text{CO}_2$  content dissolving calcite from pipe walls, previously precipitated as  $\text{CaCO}_3$  (Table 4.3).

The standard deviation decreased on the total  $\text{CO}_2$  values beginning 8/21/79, when the 2N sodium hydroxide solution was prepared from a carbonate free 50% sodium hydroxide solution. Previously, the absorbing solution had been made with reagent grade sodium hydroxide pellets. The maximum limit of impurity in the pellets according to listed specifications was 1% sodium carbonate, whereas the 50% sodium hydroxide solution had a maximum sodium carbonate impurity of <0.1%.

TABLE 4.1. Test Inlet Brine Chemistry

Parameter	Average <sup>(a)</sup>	Standard Deviation	Range	
			High Value	Low Value
pH	6.0	0.13	6.3	5.8
Conductivity $\mu$ mhos	6534	225	6848	6100
Total Dissolved Solids mg/l	4220	34	4273	4142
Alkalinity $\text{HCO}_3^-$ mg/l	575.1	21.7	601.3	530.9
Total $\text{CO}_2$ mg/l	1110	114	1332	920
$\text{SiO}_2$ mg/l	219	18	246	198
$\text{SO}_4^-$ mg/l	161.3	1.8	164	159
$\text{F}^-$ mg/l	3.4	0.2	3.9	3.0
$\text{Cl}^-$ mg/l	1937	21.4	1968	1906
$\text{NH}_3$ mg/l	14.4	1.4	17.2	11.8
Al mg/l	<0.5	0	<0.5	<0.5
As mg/l	0.6	0.1	0.7	0.4
B mg/l	7.5	0.1	7.8	7.4
Ba mg/l	0.3	0	0.3	0.3
Ca mg/l	9.4	1.4	13.1	7.6
Fe mg/l	0.1	0.07	0.3	<0.1
K mg/l	107	5.5	120	100
Li mg/l	5.4	0.16	5.6	5.0
Mg mg/l	0.4	0.06	0.5	0.3
Na mg/l	1385	18	1426	1333
P mg/l	<0.1	0	<0.1	<0.1
Si mg/l	103.9	1.4	106.1	102.2
Sr mg/l	2.2	<0.1	2.3	2.2

(a) Average of 13 days @ 3 samples/day = 39 samples.

TABLE 4.2. Loop Conditions at Time of Sampling Port 3

Date	Pressure at P-2 (psi)		Flow Gauge (gpm)	Acidification
	Data Logger	Gauge		
226-8/14		125	110	
227-8/15	126.2	125	95	
228-8/16	125.8	125	95	
229-8/17		125	85	
230-8/18	121.2	122	112	X
231-8/19	124.8	123	105	
232-8/20	132.2	135	76	X
233-8/21	131.6	129	68	
234-8/22	128.4	125	76	X
235-8/23	131.6	125	65	X
236-8/24		132	68	
237-8/25		128	69	
239-8/27		125	65	X
240-8/28		130	50	X

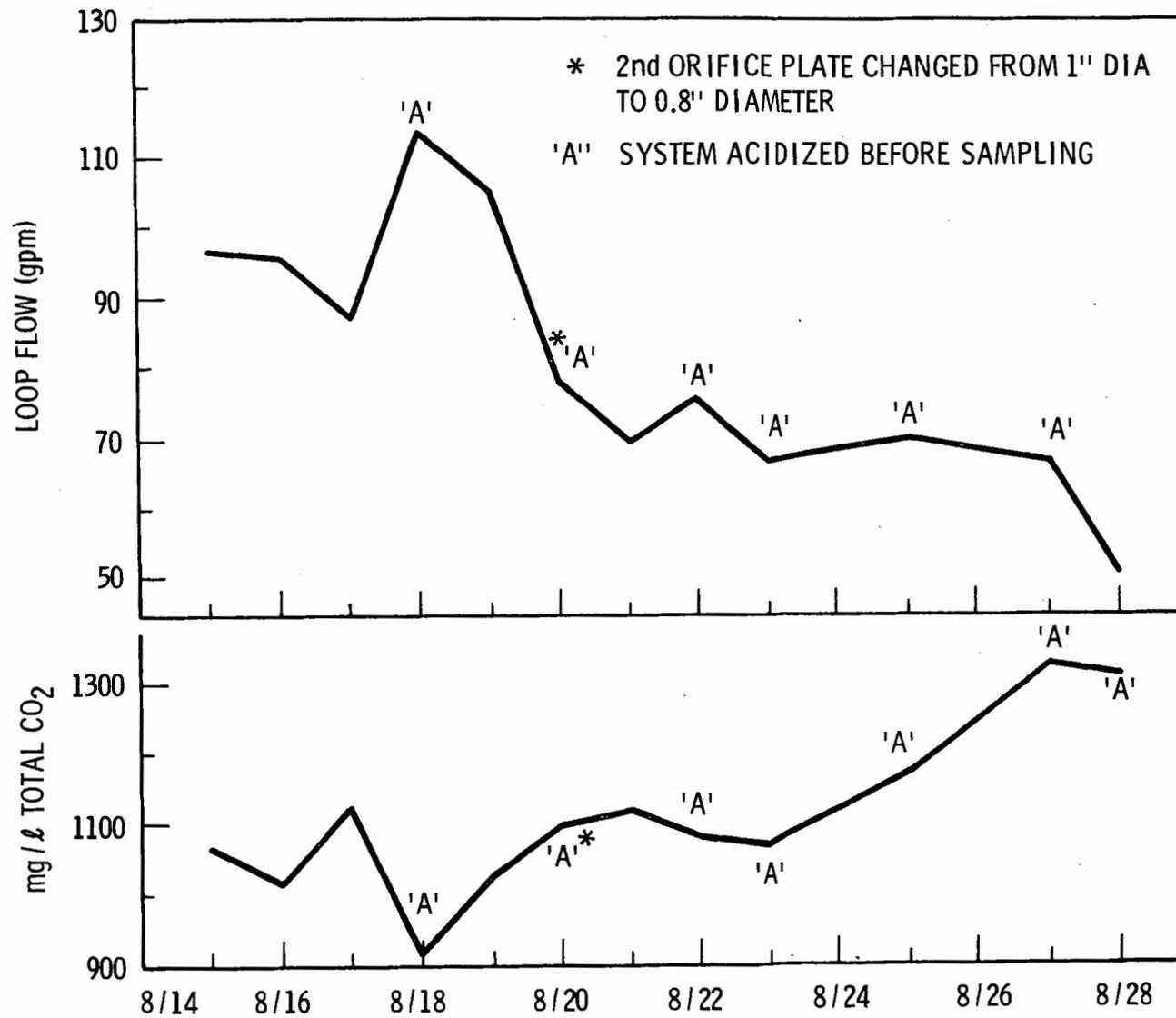
The brine composition stayed the same (within 5%) between the wellhead and the test loop inlet for all parameters throughout the test except for the total carbon dioxide and calcium values. At the onset of the testing period, values for both total CO<sub>2</sub> and calcium were higher at the wellhead than at the test inlet. At the end of the testing period, the total CO<sub>2</sub> and Ca<sup>++</sup> values had increased at the test inlet to correspond with those values at the wellhead (see Figure 4.1 and Table 4.3). This would appear to be a general trend toward an equilibrium of CaCO<sub>3</sub> scale, Ca<sup>++</sup>, and CO<sub>2</sub> in the pipe between the wellhead and the test loop during the two week testing period.

TABLE 4.3. Variation in CO<sub>2</sub> and Calcium at Test Loop Inlet

<u>Date</u>	<u>Sample Description</u>	<u>pH (a)</u>	<u>mg/ℓ Total CO<sub>2</sub> (b)</u>	<u>mg/ℓ Ca (b)</u>
227-8/15	Wellhead	5.9	1219 ± 41	10.7 ± 0.15
227-8/15	Test Loop Inlet	6.1	1065 ± 67	8.3 ± 0.11
228-8/16	Test Loop Inlet	5.9	1021 ± 56	8.3 ± 0.12
229-8/17	Test Loop Inlet	6.1	1126 ± 29	8.7 ± 0.25
230-8/18	Test Loop Inlet	6.0	920 ± 23	7.8 ± 0.21
231-8/19	Test Loop Inlet	6.1	1030 ± 50	8.6 ± 0.15
232-8/20	Test Loop Inlet	6.3	1101 ± 24	9.8 ± 0.15
233-8/21	Test Loop Inlet	6.0	1127 ± 26	9.4 ± 0.25
234-8/22	Test Loop Inlet	6.2	1087 ± 29	9.7 ± 0.34
235-8/23	Test Loop Inlet	5.8	1076 ± 16	9.6 ± 0.20
236-8/24	Test Loop Inlet	6.0	1129 ± 17	9.3 ± 0.20
237-8/25	Test Loop Inlet	6.0	1177 ± 6	13.1 ± 0.55
239-8/27	Test Loop Inlet	5.9	1332 ± 31	9.4 ± 0.10
240-8/28	Test Loop Inlet	6.0	1320 ± 38	10.8 ± 0.23
240-8/28	Wellhead	5.9	1319 ± 41	10.2 ± 0.4

(a) pH value measured at time of sampling.

(b) Average and Standard Deviation of triplicate samples taken daily.

FIGURE 4.1. Loop Flow and Total CO<sub>2</sub> Comparison

## 5.0 MEASUREMENT OF pH (TEST 3)

E. M. Woodruff and D. W. Shannon

### 5.1 INTRODUCTION

Test 3 was designed to study pH measurements under field conditions in an effort to demonstrate how some variables, which may or may not be controlled, influence results. Normally, a portable pH meter is taken into the field, calibrated with buffers and then used to measure pH by inserting a probe into geothermal fluid that has been passed through cooling coils and collected in a beaker. Fluid temperature is also usually reported. These methods generally produce inexact data because they do not measure pH levels under actual operating temperatures and pressures characteristic of geothermal systems. Little has been reported about the combined effects of temperature and gas release associated with dropping to atmospheric pressure. In this test, we installed special in-line probes and controlled the flow of a sampling system similar to that used in Test 2. Varying the temperature permitted us to examine effects over a wide range of field conditions.

### 5.2 OBJECTIVE

The purpose of this test was to establish a basis for selecting the best procedures for measuring pH of geothermal fluids in the field.

### 5.3 SAMPLING POINT

The standard 1/4-inch diameter probe used in Tests 1 and 2 was inserted through Port S-16 and adjusted so that it sampled from the center of the 4-inch loop pipe. This position is downstream from the first orifice and is, therefore, in a region where some CO<sub>2</sub> is breaking out. A second probe supplied a high temperature, high pressure, pH system based on the Leeds and Northrup in-line cell (Port S-7, located upstream of the first orifice, sampled brine at higher pressure with less gas break-out).

#### 5.4 MEASUREMENT METHODS

Our equipment arrangement, which included sensing pH electrodes, their associated meters, and the brine sampling line, is shown in Figure 5.1. Table 5.1 identifies specific items of equipment in greater detail.

Our measurement procedure involved first calibrating the electrodes in pH 7 and pH 4 buffers, then assembling them on a pegboard stand adjacent to the test loop (see Figure 5.1). We used the regulating valve to adjust the flow so that it would enter the exit-point beaker at approximately 10°C. Each meter reading was then recorded without any adjustment to the temperature compensation knob. This procedure was repeated for measurements at 20, 30, 40, 50 and 60°C. Temperatures were increased by increasing flow through the cooling coils again with adjustment of the regulating valve.

We repeated the pH readings of 40, 50 and 60°C, adjusting the compensation knob on each meter for each temperature.

TABLE 5.1. Equipment For pH Field Test

<u>Probe</u>	<u>Location/Condition</u>	<u>Electrode</u>	<u>Meter</u>
A	Port S-7•85°C, 120 psig.	L&N	Data Logger
B	In Sample Line S-16 (Upstream of Regulating Valve•Cooled, Pressurized.	Extech Model 150C Sealed Reference With 1/2" On-Stream Flow Cell	Extech Model 609
C	In Beaker•Cooled, Depressurized (Original Flow Through Plan in Figure 5.1 Aborted by Defective Probe).(a)	Orion Combination Electrode No. 910200	Orion 407A/F
D	In Collection Beaker•Cooled, Depressurized.	Markson Combination pH/Reference Model VM-830B	Markson VM-80
E	Beaker in Field Lab•Cooled, Depressurized.	Orion Combination	Orion 801

(a) The probe for cold flow-through could not be calibrated in the field, so the standard Orion probe was substituted and used in the calibration beaker.

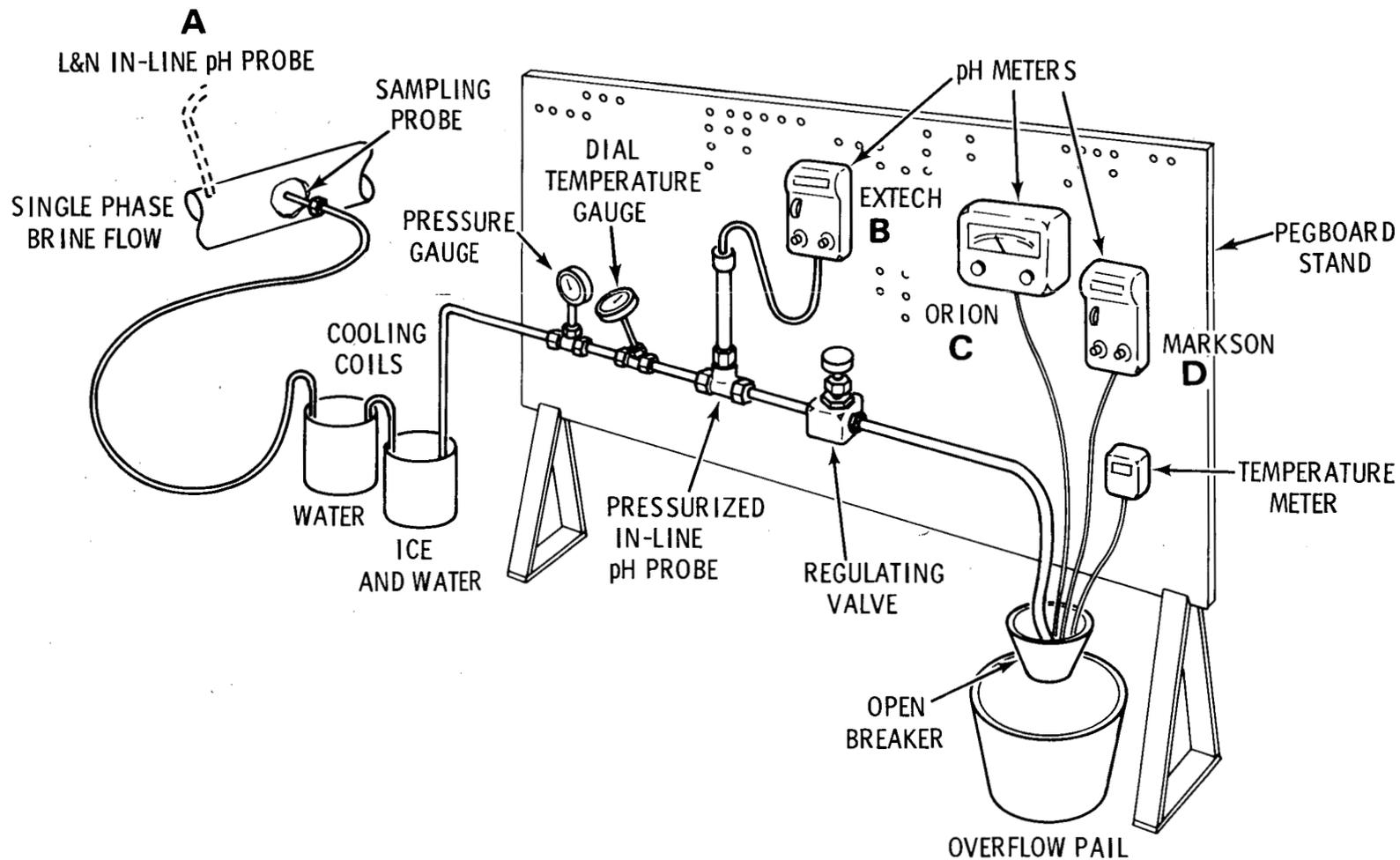


FIGURE 5.1. Field pH Test Arrangement

## 5.5 RESULTS

Measurements are tabulated in Table 5.2 and plotted in Figure 5.2.

The Orion field meter (Probe C) gave the most linear results. At 22°C, the maximum difference between meters, excluding Probe B, was 0.15 pH units. At 60°C, this spread was 0.30 for both compensated and uncompensated readings. Use of the compensating knob on meters becomes significant only for temperatures of 50°C and above.

The in-line Probe A, which operated at system pressure, but cooled to 85°C, gave constant pH readings of 5.95 to 5.97. This probe was immersed into single-phase brine that had been drawn off the bottom of a pipe located upstream from the first orifice. The total CO<sub>2</sub> in the brine was measured at 1174 mg/ℓ with HCO<sub>3</sub><sup>-</sup> at 596 mg/ℓ. An approximate pH (neglecting activity coefficients) can be calculated by:

$$\frac{[\text{H}^+][\text{HCO}_3^-]}{[\text{CO}_2(\text{ag}) + \text{H}_2\text{CO}_3]} = K_1 \quad \text{Log } K_1 \approx -6.35 \text{ @ } 85^\circ\text{C}$$

$$[\text{CO}_2(\text{ag}) + \text{H}_2\text{CO}_3] = [\text{CO}_2]_{\text{total}} - [\text{HCO}_3^-]$$

$$[\text{H}^+] = \frac{K_1 [\text{CO}_2(\text{ag}) + \text{H}_2\text{CO}_3]}{[\text{HCO}_3^-]}$$

$$\text{pH} = -\text{Log} [\text{H}^+] = 6.35 - \text{Log}\left(\frac{1,174}{44,000} - \frac{596}{61,000}\right) + \text{Log}\left(\frac{596}{61,000}\right)$$

$$\text{pH} = 6.1$$

The near agreement between the measured pH (5.96) and the calculated pH (6.1) is excellent, given that so many variables other than CO<sub>2</sub> can affect the pH, and that the HCO<sub>3</sub><sup>-</sup> value was taken from a depressurized sample.

The total CO<sub>2</sub> and HCO<sub>3</sub><sup>-</sup> content of the gas/liquid mixture sampled from Port 16 tended to fluctuate because the port is located just downstream of the first orifice. One measurement made from Port 20 on the same day showed a total CO<sub>2</sub> content of 5,921 mg/ℓ. Calculations based on this latter value showed a 30°C pressurized, cooled brine pH of 5.2 (Log K<sub>1</sub> = -6.33 at 30°C).

TABLE 5.2. Test 3 pH Measurements

A		B		C		D		E
Hot, Port 7	Cooled Probes	Cold Extech. Pressurized (112 psig)		Field Orion. Cold, Depressurized		Markson. Cold, Depressurized		Lab Orion. Cold, Depressurized
pH/ Fluid Temp. °C	Nominal Fluid Temperature °C	Temperature Compensated pH/ Fluid Temp. °C	Temperature Uncompensated pH/ Fluid Temp. °C	Temperature Compensated pH/ Fluid Temp. °C	Temperature Uncompensated pH/ Fluid Temp. °C	Temperature Compensated pH/ Fluid Temp. °C	Temperature Uncompensated pH/ Fluid Temp. °C	Temperature Uncompensated pH/ Fluid Temp. °C
5.97/84.7	10		5.34/11.9		5.62/11.8		5.7/12.2	5.694/--
5.97/86	20		4.70/19.7		5.75/22		5.6/22	5.579/22
5.96/85.9	30		5.08/29.8		5.87/29.8		5.6/29.8	5.594/30
5.95/86	40	5.0/40	4.74/42	6.0/40	6.0/40	5.7/40	5.7/41	5.790/--
5.97/85	50	4.82/50.4	4.63/49.1	6.15/50	6.1/49.3	5.8/50	5.7/49.8	5.74/--
5.95/85.4	60	5.25/60.8	4.93/59	6.2/60.4	6.1/59.2	5.9/60.0	5.8/59.6	6.025/60

5.6

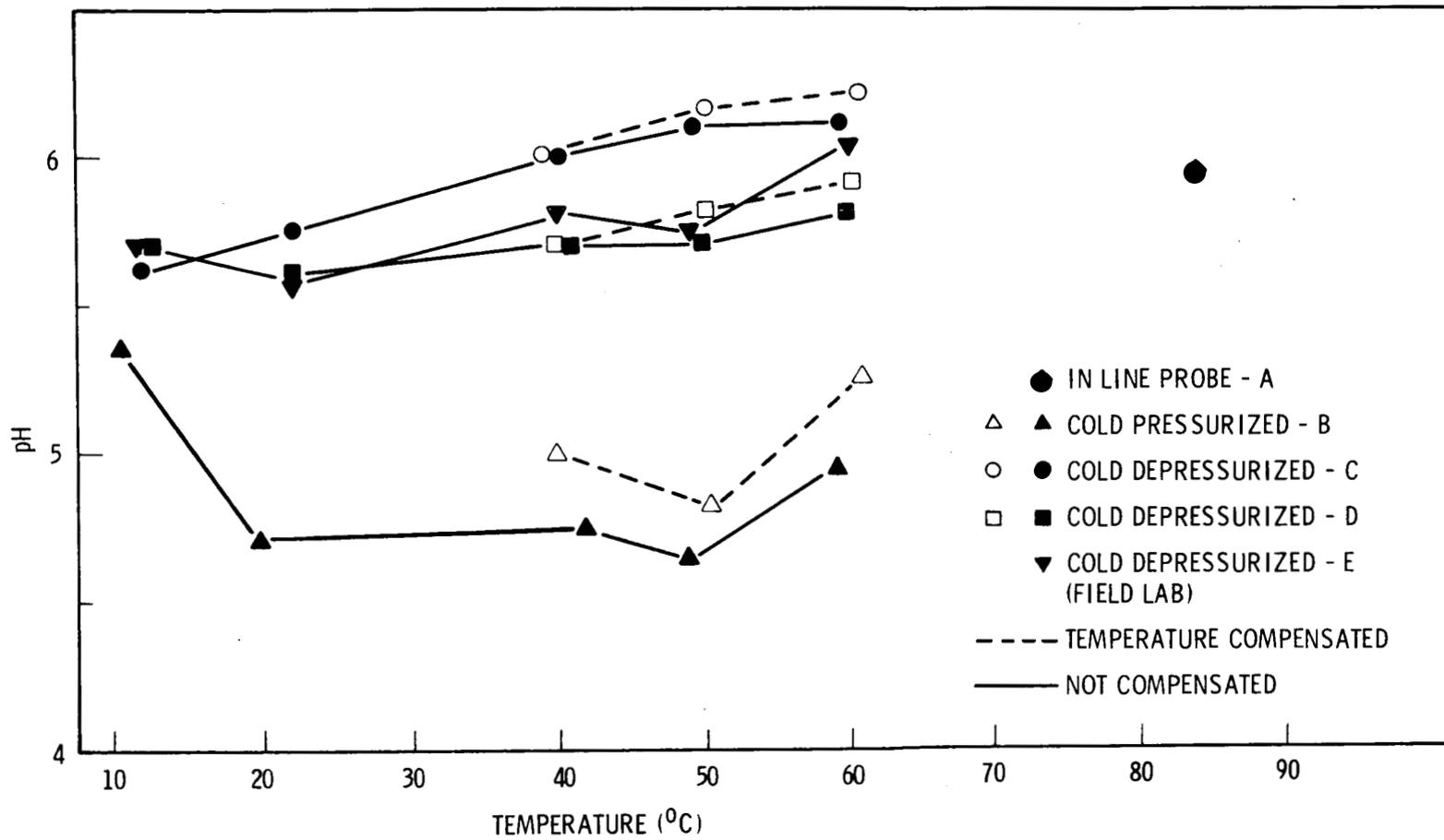


FIGURE 5.2. pH Probe Test Results

The measured values in the 10 to 40°C range were 4.7 to 5.3. This apparently small change in K suggests that total CO<sub>2</sub> varied significantly. In any event, the pressurized, cooled sample should give the lowest reading. Our data support this tendency.

The various depressurized samples show higher pH values of 5.6 to 6.0, reflecting loss of the gaseous CO<sub>2</sub> on depressurization. The residual CO<sub>2</sub> content of this liquid was not measured, but should have been slightly more than the 1,174 mg/l measured at Port 3 since the liquid was still bubbling with CO<sub>2</sub> release. Using an estimated value of 1,500 mg/l CO<sub>2</sub> we calculate a pH of 5.9.

Following the sample depressurization, pH measurements are very time-dependent. Values rise sharply immediately after the CO<sub>2</sub> is released, then increase incrementally as the rest of the CO<sub>2</sub> is released over a period of several days. This can be seen in the data in Table 5.3. Using sets of brine-filled bottles, we measured pH levels at various intervals of time. Our data indicate that there is little change in pH in the first 30 min after depressurization but significant drift over the next few days.

## 5.6 CONCLUSIONS

No pH measurement of a cooled sample will be representative of the actual pH at high temperatures unless corrections are made for various acid-base equilibria. We see a real need for installing high temperature, high pressure pH electrodes in the in-line system. (Such electrodes are under development at PNL). Currently available commercial electrodes with a temperature limit of 100°C, and a pressure cell at 100°C, or a pressure cell on a coil outlet of 25°C, provide the least ambiguous pH of all electrodes currently available. However, both of these cells are very complex. If, on the other hand, a simple arrangement is used, the pH of a sample at the coil outlet can be measured at atmospheric pressure within the first 15 min. If pH is measured at 1 atm pressure at 25°C, a notation to this effect alerts others that they can use the total CO<sub>2</sub> analysis to correct the pH value to loop pressure and temperature.

TABLE 5.3. pH Change in Stored Brine Over Time

<u>Storage Time of Brine Before Measurement</u>	<u>pH</u>
0	5.58
15 min.	5.65
30 min.	5.46
1 day	5.82
10 days	6.13

## 6.0 SAMPLING FOR TOTAL CARBON DIOXIDE CONTENT

M. Cole and C. H. Kindle

### 6.1 INTRODUCTION

We can maximize the energy efficiency of using geothermal heat for electric power production if we have accurate knowledge of  $\text{CO}_2$  content. The quantity of  $\text{CO}_2$  present will influence turbine design for flash power production plants. The  $\text{CO}_2$  content also influences carbonate scaling, which can be a major problem in certain geothermal reservoirs.

This chapter deals with seven different sampling and collection techniques used to contain  $\text{CO}_2$  for subsequent analysis including sparging, evacuated bulb, and separator variations.

The methods are:

- Straight Bottle Sparge
  - Single
  - Double
- Double Tube Sparge
- Fisher Milligan Absorption Bottles
- Cerro Prieto (Mexican 5ℓ Evacuated Flask)
- Truesdell (small evacuated flask reported by A. H. Truesdell)<sup>(1)</sup>
- Bottle Sparge With Separator

During this comparison, we identified three facets that all of the methods have in common: 1) they use precooled fluids; 2) they involve pressure reduction; and 3) they use NaOH to trap  $\text{CO}_2$ . NaOH is used because it reacts with both dissolved and gaseous  $\text{CO}_2$ . Volumetric gas sampling schemes work solely on the gaseous fraction.

The fluid was precooled to about  $25^\circ\text{C}$  at the head of the sampling system (Figure 3.2). Precooling accomplished three ends: 1) it prevented steam

flashing during pressure release; 2) it made the mechanics of sampling easier because it eliminated high temperatures, and 3) it increased the solubility of  $\text{CO}_2$  in the brine. Reducing pressure with the use of valves simplifies the equipment and makes subsequent data handling and analysis easier. The  $\text{CO}_2$  trapping method, a classical method of containing  $\text{CO}_2$  for subsequent analysis, is demonstrated by the following equation:



## 6.2 CONCLUSIONS

The simplest and least expensive sampling and collection technique, single-bottle sparging with full cooled flow into NaOH solution, was found to be accurate to within a few percent of total  $\text{CO}_2$ . This sparging method is independent of the gas/liquid ratio, and we recommend it for use on flows containing less than 5,000 mg  $\text{CO}_2$  per liter of brine.

Other methods yielded comparable results, however and may be preferable to single-bottle sparging if the combined sampling rate and suspected  $\text{CO}_2$  concentration are high enough to overwhelm the single sparge trap. The addition of a second sparge bottle in series helps to accommodate higher volumes. The other five methods we tested proved to be too awkward or fragile for convenient use in the field.

The most precise data were produced whenever we preweighed the bottles of NaOH absorbing solution and reweighed them after collecting the sample. However, the precision of both volumetric and the gravimetric determinations were better than +5%. (Time is saved if one measures 500 ml of NaOH solution into the bottle and then fills it to a previously made 1,000 ml mark with the brine +  $\text{CO}_2$ .)

No advantage was found by separating the gas from the liquid and then collecting  $\text{CO}_2$  from the respective gas and liquid streams. Trapping the  $\text{CO}_2$  from the mixed liquid/gas flow was just as effective.

When sampling two-phase flow, it is almost certain that the ratio of gaseous  $\text{CO}_2$  to liquid will not be typical of unflashed brine. Depending on the

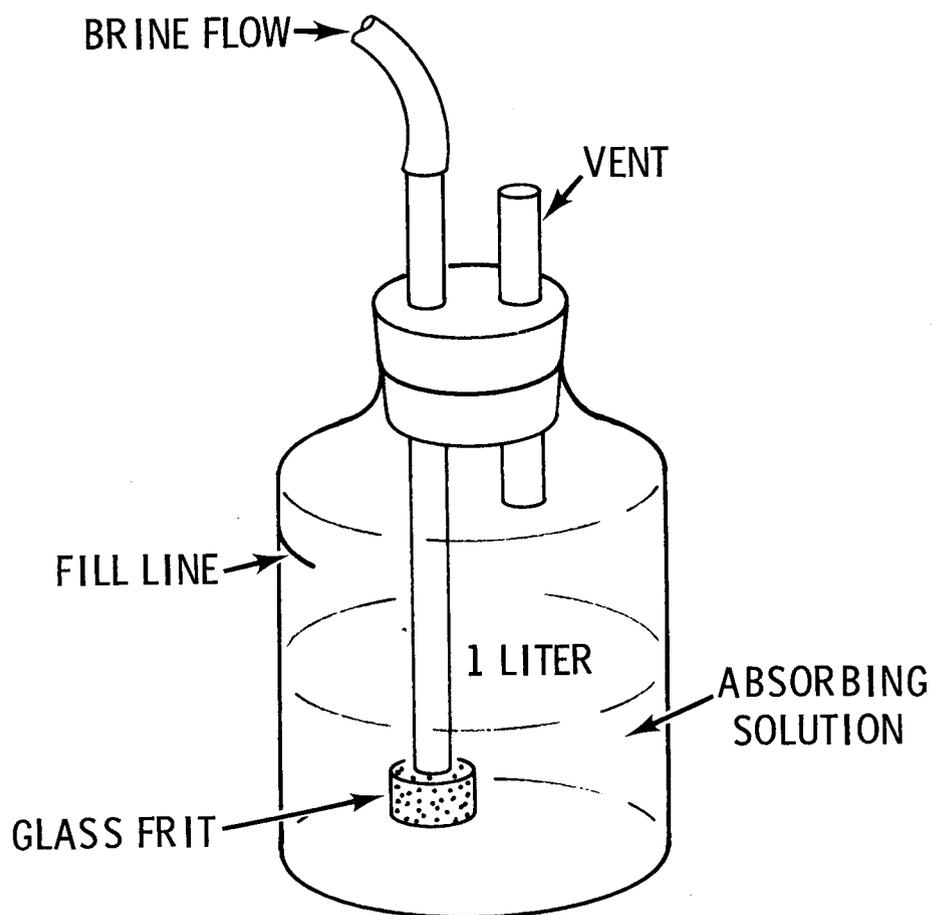
gas/liquid ratio, the total  $\text{CO}_2$  value can vary by a factor of as much as 10,000, because of nonhomogeneity in the pipes. Taking a representative sample from an inhomogeneous two-phase flow remains a problem (see Sections, 10, 11, 12).

#### 6.2.1 Total $\text{CO}_2$ Sampling Methods

Seven different sampling methods or variations for absorbing  $\text{CO}_2$  into a NaOH solution were used for this study and are illustrated in Figures 6.1 through 6.7. All of the collection containers were weighed prior to the addition of 2N NaOH absorbing solution, after which they were reweighed. A third weight was obtained after the gas or brine sample was collected. Thus, the weights of the NaOH and the brine are known.

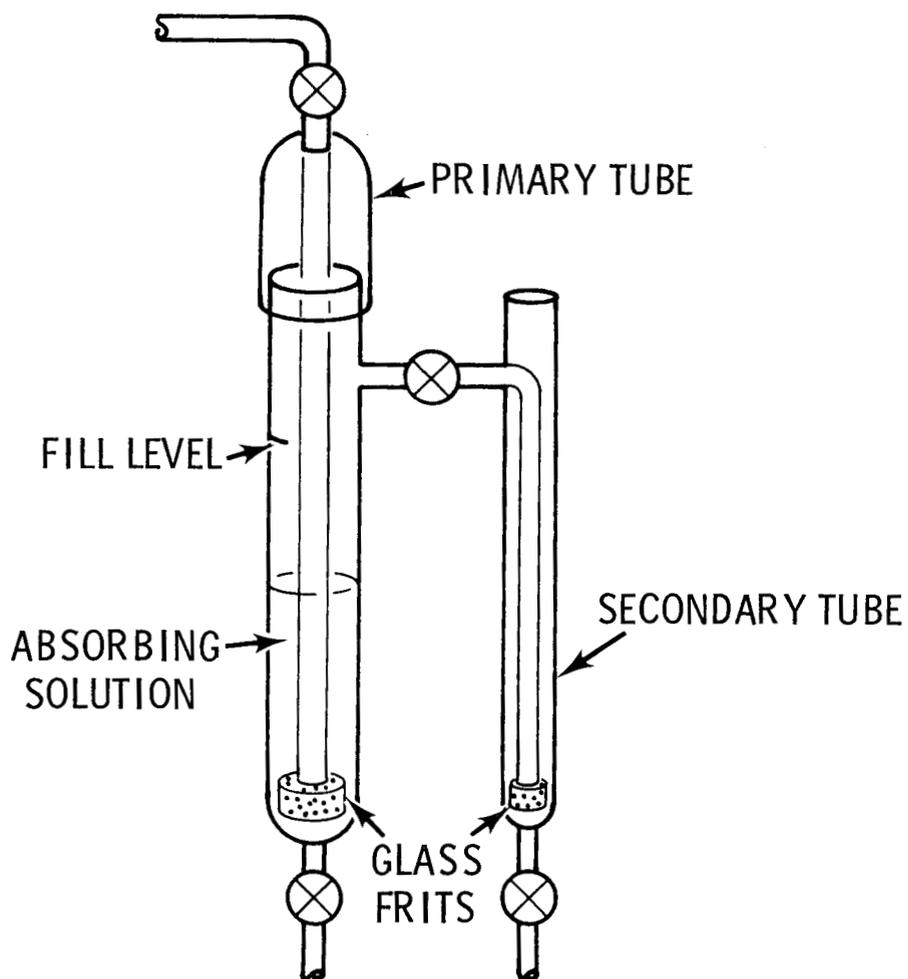
In all cases, before allowing a pressure drop to occur, the brine was cooled. Samples were collected in triplicate at three ports of the test loop (see Figures 1.2 and 3.2). Port 16 is a horizontal port in the  $\text{CO}_2$ - and water-region of the two-phase test loop. Ports 56 and 76 are vertically located in the two-phase steam-gas-and-water section; Port 76 is located the furthest downstream from the orifice.

Shown in Figure 6.1 is the single sparge method. The sample flows through a gas dispersion tube inserted into 500 ml of NaOH absorbing solution. The bottle has a calibration mark at 1 liter to which point 500 ml of brine is added. Figure 6.2 illustrates the double-tube sparge method. The systems primary tube contains 500 ml of NaOH, while the smaller, secondary tube contains 100 ml. Sampling is accomplished by opening the two upper stopcocks and allowing the brine to finish filling the primary tube to the 1 liter mark. The two lower stopcocks are used to transfer the samples to storage bottles. Any unabsorbed  $\text{CO}_2$  escaping from the large volume is collected in the secondary tube. Figure 6.3 demonstrates the Fisher Milligan method. In this design, 100 ml of NaOH is added to the first bottle and 200 ml to the second. Only 100 ml of brine is then collected in the first bottle. The fourth method (Figure 6.4) utilizes a Cerro Prieto flask, which is patterned after a method used in New Zealand. After the addition of 500 ml of NaOH, the flask is evacuated to the practical limit of the vapor pressure of water. During



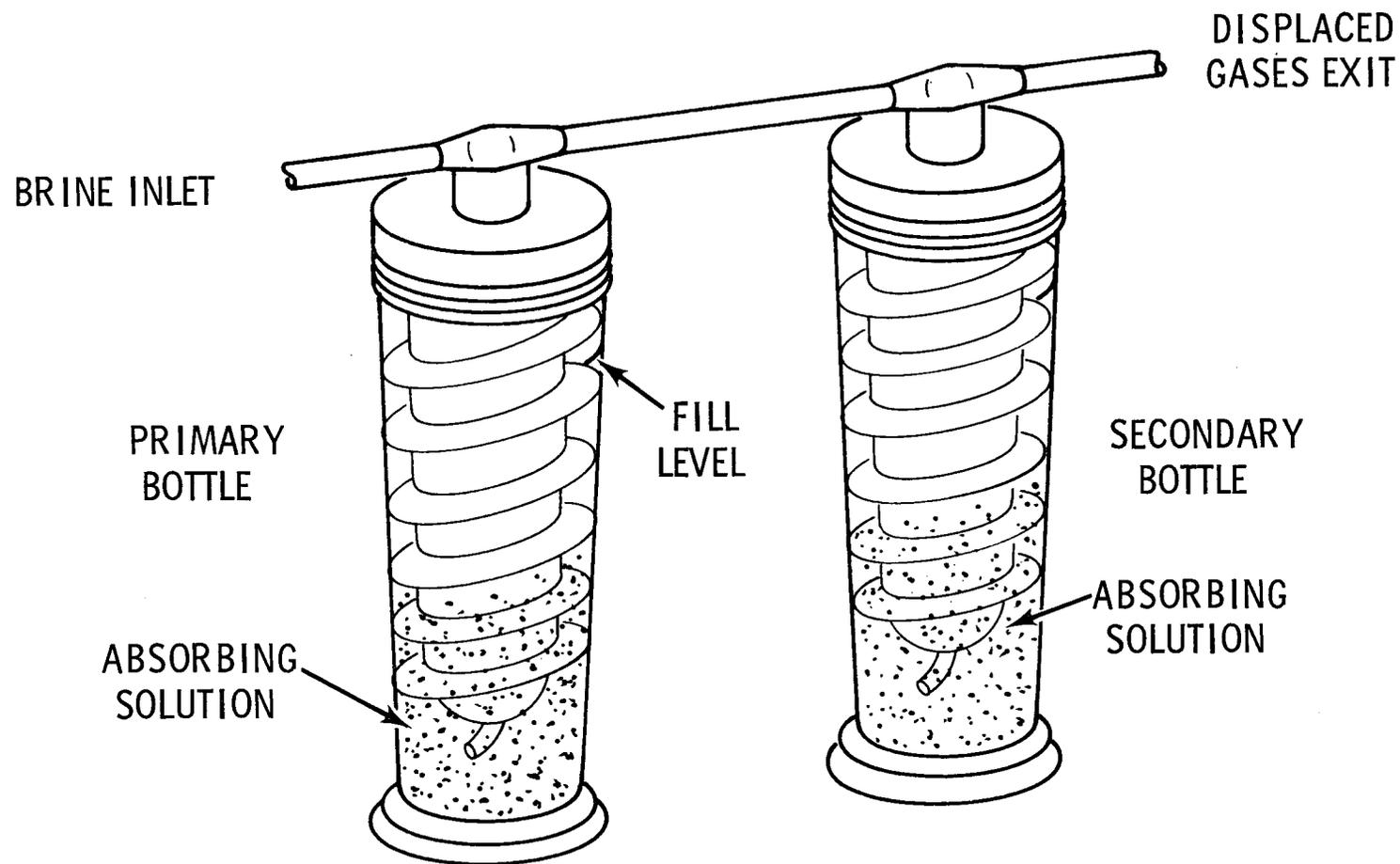
THE BRINE IS SPARGED DIRECTLY INTO 500 ml OF 2N NaOH. ENOUGH BRINE IS ADDED TO BRING THE TOTAL VOLUME TO 1 LITER.

FIGURE 6.1. Single Bottle Sparge



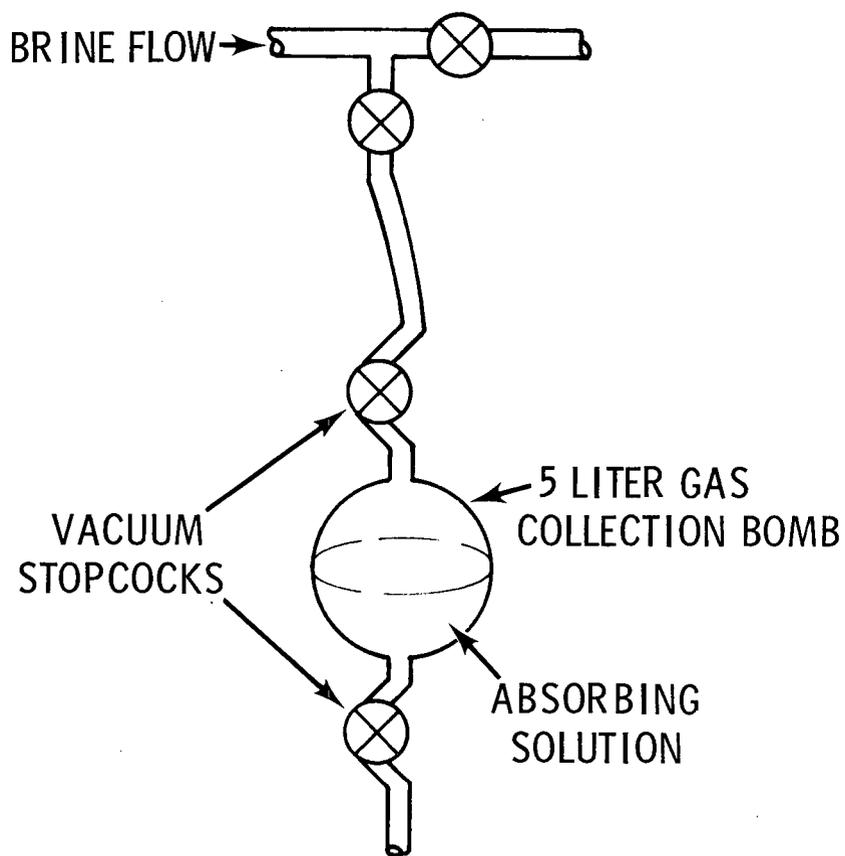
500 ml OF 2N NaOH IS ADDED TO THE PRIMARY TUBE AND 100 ml TO THE SECONDARY TUBE. THE BRINE IS SPARGED INTO THE PRIMARY TUBE TO A TOTAL VOLUME OF 1 LITER. THE SECONDARY TUBE ABSORBS THE EXCESS GAS.

FIGURE 6.2. Double-Tube Sparger



100 ml OF 2N NaOH IS ADDED TO BOTH BOTTLES. THE FILL LEVEL OF THE PRIMARY BOTTLE IS 200 ml.

FIGURE 6.3. Fisher Milligan Method



500 ml OF 2N NaOH IS PLACED IN THE FLASK, WHICH IS THEN EVACUATED. FOR SAMPLING, THE APPROPRIATE STOPCOCKS ARE OPENED AND THE PRESSURES ALLOWED TO EQUALIZE.

FIGURE 6.4. Cerro Prieto Method

sampling, enough brine is allowed to enter to equalize the pressures between the sampling line and the flask. Another evacuated-flask method, the Truesdell method, is described by Figure 6.5. For this method, approximately 300 ml of the sample is collected in 100 ml of absorbing solution. The brine is drawn into the evacuated bottle when the hole in the collection tube is slid inside the O-rings. Figure 6.6 shows the double-bottle sparge method a simple modification of the single-sparge method. Except for the addition of the second bottle, which also contains 500 ml of absorbing solution, the sample collecting methods are identical. A further modification of this method is demonstrated in Figure 6.7. The double-bottle sparge method is used to sample the liquid from the condensate line of a steam separator. A glass gas sampling level is used to collect the noncondensables as described in Section 10.

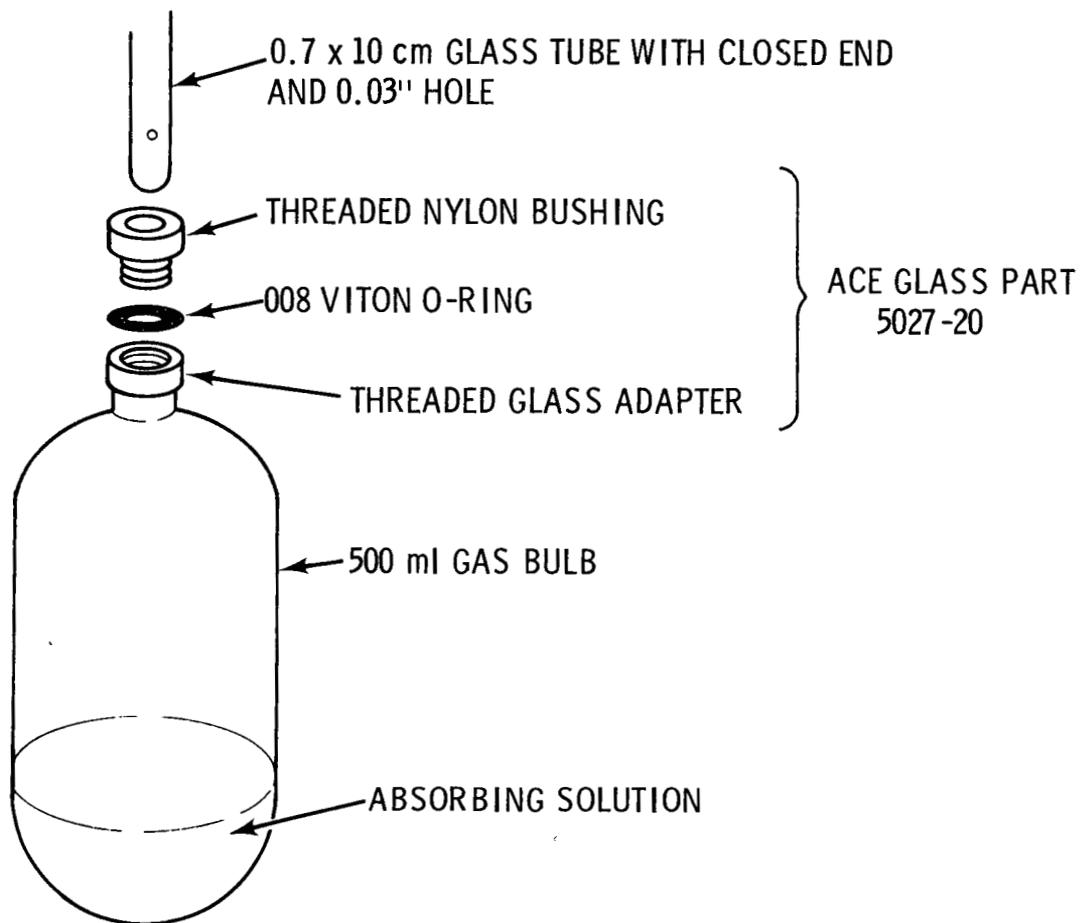
### 6.2.2 Results

All of the sparge methods give comparable values (see Table 6.1) for the total CO<sub>2</sub> content, however, not without some experimental difficulties. Generally, the one- and two-bottle sparge methods are the most convenient and free of experimental and operator problems.

The bottle sparge methods collect the samples directly in the storage bottles while both the double-tube sparge and the Fisher Milligan methods necessitate the transfer of the samples once they are collected. In addition to this relocating inconvenience, the possibilities for operator contact with the NaOH and of sample contamination increase, and a quantitative transfer may not be obtained.

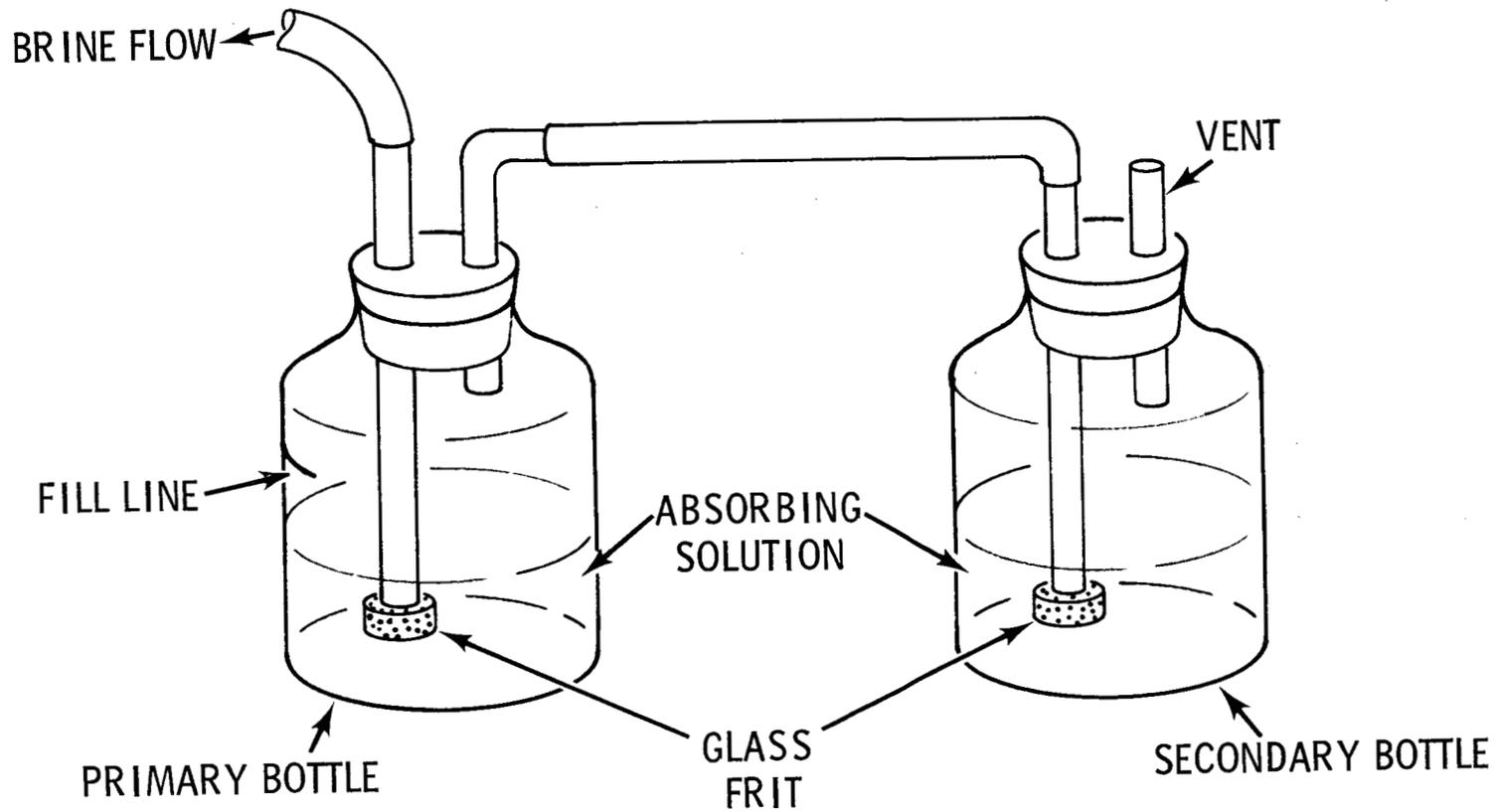
TABLE 6.1. Comparison of Three Sparge Methods For CO<sub>2</sub> Sampling  
(Total Measured at Port 16)

<u>Method</u>	<u>Total CO<sub>2</sub> (mg CO<sub>2</sub>/ℓ Brine)</u>	<u>Standard Deviation</u>
Single-Bottle Sparge	2,721	+76 (2.8%)
Double-Tube Sparge	2,746	+14 (0.5%)
Fisher Milligan (a double sparge)	2,509	+677 (27%)



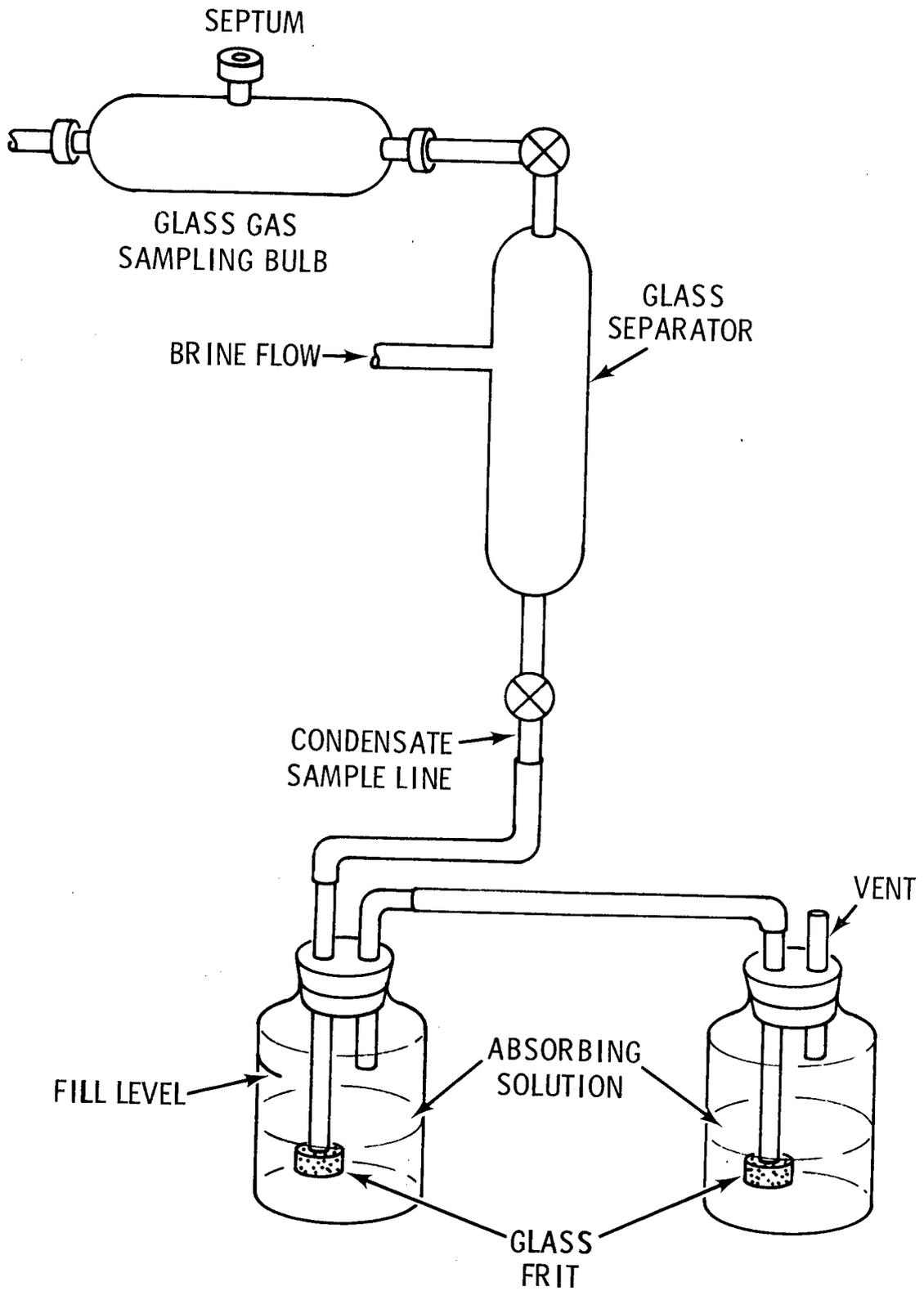
THE FLASK IS EVACUATED WITH 100 ml 2N NaOH. SAMPLING IS ACCOMPLISHED BY PRESSURE EQUALIZATION. THE BOTTLE IS OPENED AND CLOSED BY SLIDING THE HOLE OF THE GLASS TUBE ON THE APPROPRIATE SIDE OF THE O-RING.

FIGURE 6.5. Truesdell Flask



500 ml OF 2N NaOH IS PLACED IN BOTH BOTTLES. 500 ml OF BRINE IS ADDED TO THE PRIMARY BOTTLE. THE SECONDARY BOTTLE ABSORBS ANY CO<sub>2</sub> ESCAPING THE FIRST.

FIGURE 6.6. Double Bottle Sparge



**FIGURE 6.7.** Sparge With Separator

When solution weights are the basis for CO<sub>2</sub> calculations, any unknown, unaccounted for solution left in the collection containers will decrease the accuracy and precision of the results because the final weight obtained is that of the drained solution only.

The large double-tube sparge is the most complicated to operate of these sparge methods because two stopcocks and a valve must be opened simultaneously to initiate sampling. Also, a large amount of unwieldy glassware must be kept clean, which is sometimes difficult to do in the field.

It would appear that the most precise method in Table 6.1 is the double-tube sparge. However, the standard deviation for this method was calculated using only two values instead of three: one reading had to be dropped because of a sampling error. The average value for the Fisher Milligan method may be only coincidentally similar to the other methods. These samples were taken during a violent thunderstorm, which may have influenced a drop in the CO<sub>2</sub> concentration from 3,200 for the first sample to 1,900 for the third sample.

The results of both of the evacuated bulb methods (see Figures 6.4 and 6.5) are hard to compare to each other and to the sparge methods (see Table 6.2) because the head space in the evacuated bulb tests contained mostly air. In addition, only two of five samples survived from the field for analysis.

We also investigated the effect the gas-to-liquid ratio had on CO<sub>2</sub> analytical accuracy. A steam separator was used to allow the separate collection of each phase from the flashed region in the test loop. Table 6.3 gives the results of the subsequent analyses and a comparison with an identical sample

TABLE 6.2. Comparison of Bottle Sparge and Evacuated-Bulb Sampling Methods (CO<sub>2</sub> Values From Port 76)

<u>Method</u>	<u>CO<sub>2</sub> mg/l Brine</u>	<u>Standard Deviation</u>	<u>Experimental Success Rate</u>
Bottle Sparge Methods (Single and Double)	5,662	+283 (5%)	100%
Evacuated Bulb Methods (Cerro Prieto and Truesdell)	1,619	+567 (35%)	40%

TABLE 6.3. Effect of Gas/Liquid Separation on CO<sub>2</sub> Determination (CO<sub>2</sub> Results From Port 56)

<u>Collection Method</u>	<u>Brine CO<sub>2</sub> Content (mg/ℓ)</u>			<u>Standard Deviation</u>
	<u>Gas</u>	<u>Liquid</u>	<u>(Combined) Two Phase</u>	
With Gas/Liquid Separator	14,913	2,508	17,421	+505 (2.9%)
Full Flow Without Separator	--	--	15,950	+1500 (9.4%)

collected by the double-bottle sparge method. The values are the same when compared in the light of their standard deviations.

As the CO<sub>2</sub> concentration increases in the cooled brine flow, the amount of CO<sub>2</sub> not absorbed by the first sparger increases. As shown in Table 6.4, this error increases as the total CO<sub>2</sub> content exceeds 6,000 mg/ℓ. Thus, in any unknown field, a double sparging method (Figure 6.6) should be used until the approximate CO<sub>2</sub> content is identified and verified to be below about 5,000 mg/ℓ.

Representative sampling in the two-phase region is still not feasible. Table 6.5 shows that as the gas-to-liquid ratio increases, the total CO<sub>2</sub>

TABLE 6.4. Collection Efficiency of Simple Bottle Sparge as a Function of CO<sub>2</sub> Concentration (Measured Relative to Double Bottle Sparge)

<u>CO<sub>2</sub> (mg/ℓ Brine)</u>	<u>% CO<sub>2</sub> Collected</u>
2,700	99.5
5,700	98
13,000	75

TABLE 6.5. Two-Phase Flow Inhomogeneities as Evidenced by Gas Changes in Sample Stream

<u>Test Loop Position</u>	<u>Gas-Liquid Ratio (lg/ℓ Brine)</u>	<u>CO<sub>2</sub> (mg/ℓ Brine)</u>
16 (+1")	0.35	2,700
76 (+4")	1.1	5,700
56 (+3")	8.7	17,000

concentration also increases. Since the true value of this brine is 1,100 mg CO<sub>2</sub>/ℓ brine, it is obvious that a disproportionate amount of gas, high in CO<sub>2</sub> content, is being drawn off the test loop by the sample probe inserted into the two-phase region.

In summary, the double-bottle sparge method is recommended for use unless the CO<sub>2</sub> concentration is known to be less than 5,000 mg/ℓ (see Table 6.6 and Figure 6.6). In this case, the single-bottle sparge is sufficient.

### 6.2.3 Analytical Factors

As mentioned in the section on sampling methods, each sample was weighed three times to obtain the individual weights of the absorbing solution and the brine. Utilizing these weights also requires the use of the specific gravities of the solutions. Collecting data about specific gravities is very expensive and unnecessary for the solutions were initially measured by volume (500 ml of NaOH followed by the addition of brine, for a total of 1 ℓ). Instead, we suggest using a simple volumetric dilution factor of 1,000/500 = 2.00. Figure 6.8 shows the results for the daily sampling for CO<sub>2</sub> from Port 3. A

TABLE 6.6. Summary of CO<sub>2</sub> Sampling Methods

<u>Method (Figure Number)</u>	<u>Advantages</u>	<u>Disadvantages</u>
Single-Bottle Sparge (8.1)	Convenient, Accurate	Accommodates 5,000 mg CO <sub>2</sub> /ℓ Brine or Less
Double-Tube Sparge (8.2)	Accurate	Inconvenient, Requires Sample Transfer
Fisher Milligan (8.3)	Secondary Trap	Inconvenient, Requires Sample Transfer
Cerro Prieto (8.4)	Closed System, Ideally No Sample Loss	Inconvenient, Requires Vacuum Equipment
Truesdell (8.5)	Closed System	Inconvenient, Requires Vacuum Equipment
Double-Bottle Sparge (8.6)	Convenient, Accurate, Will Trap up to 20,000 mg CO <sub>2</sub> /ℓ Brine and Steam	Less Convenient Than Single Bottle Sparge, More Time and Analyses Required

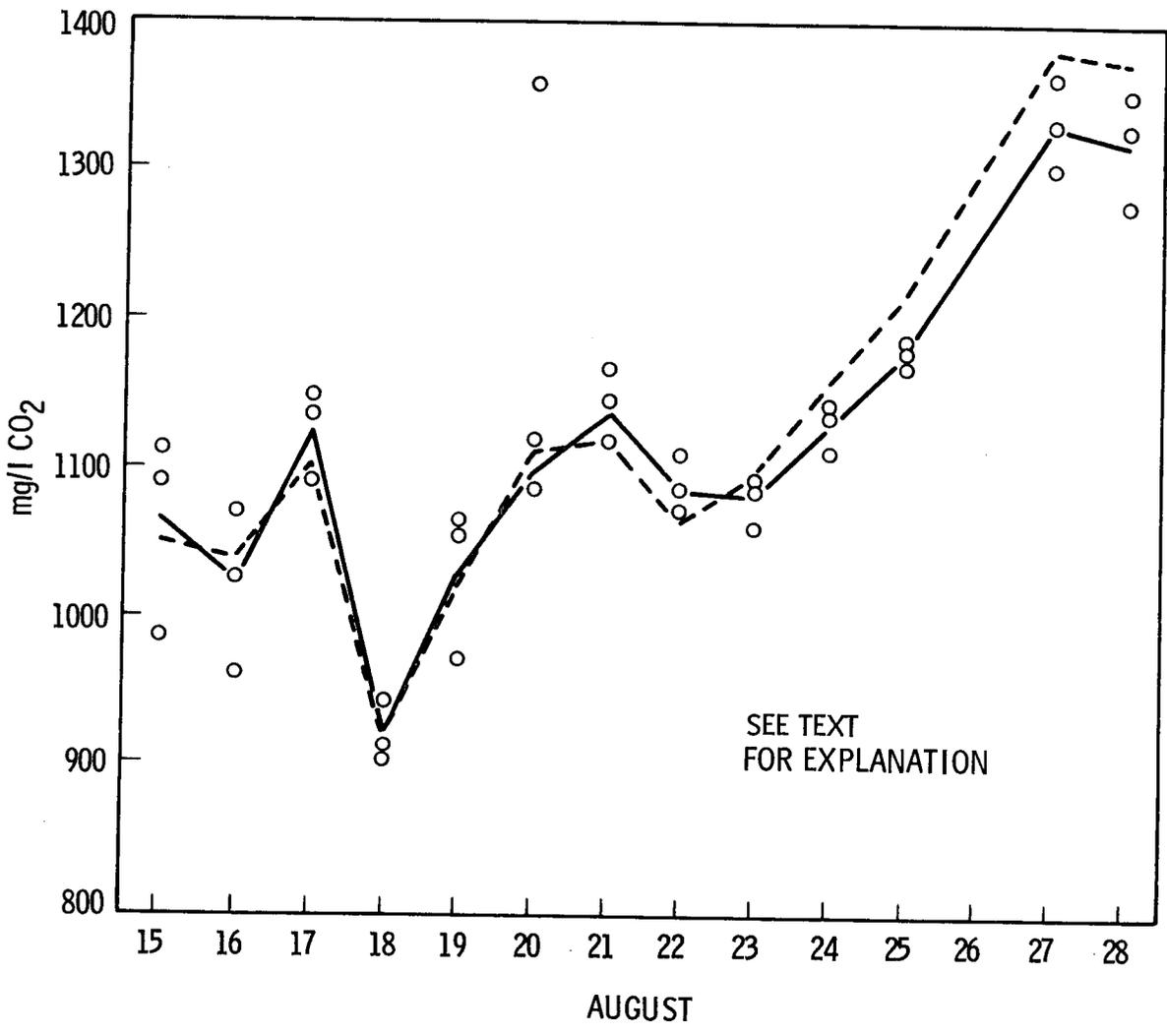


FIGURE 6.8. Volumetric vs. Gravimetric CO<sub>2</sub> Determination Using Single Bottle Sparge Method

solid line was drawn through the averages calculated using weights and densities. The dotted line represents the same data calculated with a volumetric dilution factor of 2. The volumetric method is certainly adequate.

The CO<sub>2</sub> content, using weights and densities, may be calculated with the following equations:

$$A = \frac{(\text{Eq. Wt. CO}_2)(V_{\text{HCl}})_{\text{sa}}(N_{\text{HCl}})_{\text{sa}} 1000}{V_{\text{sa}}} \quad (2)$$

$$B = \frac{(\text{Eq. Wt. CO}_2)(V_{\text{HCl}})_{\text{blk}}(N_{\text{HCl}})_{\text{blk}}(\text{Wt. NaOH})(\rho_{\text{sa}})(1000)}{(V_{\text{NaOH}})_{\text{blk}}(\rho_{\text{NaOH}})(\text{Wt. sa})} \quad (3)$$

$$\text{DF} = \frac{\text{Wt. sa}/\rho_{\text{sa}}}{\text{Wt. sa}/\rho_{\text{sa}} - \text{Wt. NaOH}/\rho_{\text{NaOH}}} \quad (4)$$

$$\text{Total CO}_2 = (V-B)(\text{DF}) \quad (5)$$

In these equations:

$$A = (\text{mg CO}_2/\ell_{\text{sa}})_{\text{sa+blk}}$$

$$B = (\text{mg CO}_2/\ell_{\text{sa}})_{\text{blk}}$$

$\rho$  = indicated density in g/ml

sa = brine and NaOH mixture

If exactly 500 ml of NaOH solution and 500 ml of brine are mixed, Equation 3 becomes:

$$B = \frac{(\text{Eq. Wt. CO}_2)(V_{\text{HCl}})_{\text{blk}}(N_{\text{HCl}})_{\text{blk}} 1000}{(V_{\text{NaOH}})_{\text{blk}}} \quad (6)$$

Equation 4 becomes:

$$\text{DF} = 2.0 \quad (7)$$

and Equation 5 becomes:

$$\text{Total CO}_2 = 2 (A-B) \quad (8)$$

These equations are only valid if exactly one equivalent of carbonate is titrated. Hydrochloric acid of an appropriate concentration is used for the titration of the sample from a pH of 8.3 to 4.5. Within this pH range, the equation is:



The titration should be done in a closed container so that the stirring does not cause  $\text{CO}_2$  absorption from the air.

## 7.0 MEASUREMENT OF H<sub>2</sub>S

E. M. Woodruff

### 7.1 INTRODUCTION

Determination of H<sub>2</sub>S dissolved in fluid samples requires either 1) stabilization in zinc acetate to prevent oxidation or loss of H<sub>2</sub>S to the air during shipment back to a central laboratory, or 2) immediate determination in the field with kits based on a color comparison developed by the methylene blue method. In either case, the ability to retain H<sub>2</sub>S in solution until the determination or stabilization takes place is influenced by the rate of gas release, which is itself influenced by temperature. Some prior experience also suggests that certain chemicals used in the field kits are temperature-sensitive, failing to develop color when employed directly on 80°C samples taken from hot springs.

### 7.2 OBJECTIVE

The purpose of this test was to compare field and laboratory results for samples taken at temperatures encountered in sampling operations. With these results, we should be able to develop guidelines for H<sub>2</sub>S sampling that improve the reliability of data and facilitate agreement between field and lab results.

### 7.3 SAMPLE POINT

Field analysis for H<sub>2</sub>S at several sampling-fluid temperatures was performed in conjunction with pH measurement experiments (Chapter 5) at sample Port S-16. Laboratory samples were taken at the same location.

### 7.4 MEASUREMENT METHODS

Field determinations were performed with CHEMetrics Sulfide Test Kit Model S-10®. This kit consists of disposable glass ampoules, which contain a measured quantity of color-forming reagent sealed under vacuum. The liquid

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®CHEMetrics, Inc., Millrun Drive, Warrenton, VA 22186.

sample is introduced by breaking the tip of the ampoule under the surface of the sample. After a prescribed period of color development (5 min for H<sub>2</sub>S kits), the ampoule is compared with standard liquid color comparators. Two comparators in the kit cover the ranges of 0.1 to 1 ppm and 1 to 10 ppm, respectively. Provisions for diluting samples of higher concentration are included in the kit in the form of a 1 ml graduated syringe, which is easy to read and effective in the field.

For these experiments, flow through the sampling cooling coils was controlled to produce temperatures of nominally 10, 20, 30, 40, and 50°C. Samples were taken for field determinations and the results recorded in field notes. Laboratory samples were taken in weighed zinc acetate solution by two methods:

- Pouring the solution into a graduate and adding sample fluid through a glass frit sparge tube until the volume was doubled. The stabilized solution was then transferred back to a sample bottle, sealed and shipped to the laboratory.
- Inserting the sparge tubes directly into the solution in the weighed bottle and filling to the 1 l mark. (500 ml of zinc acetate was added before weighing.)

## 7.5 RESULTS

The results for field and laboratory determinations are given in Table 7.1 and plotted in Figure 7.1.

The most consistent data resulted from lab results on samples sparged directly into sample bottles. A small decline in concentration from 1.45 mg/l at 20°C to 1.35 mg/l at 50°C is indicated. Erratic results for samples sparged into the graduated cylinder indicate that these attempts to improve controls on the zinc acetate/sample fluid volume relationship were unsuccessful.

Two observations on field data are important. First, it should be noted that this particular sample fluid gave lab results just above the most sensitive color comparator in the field kit. The closest comparison points for

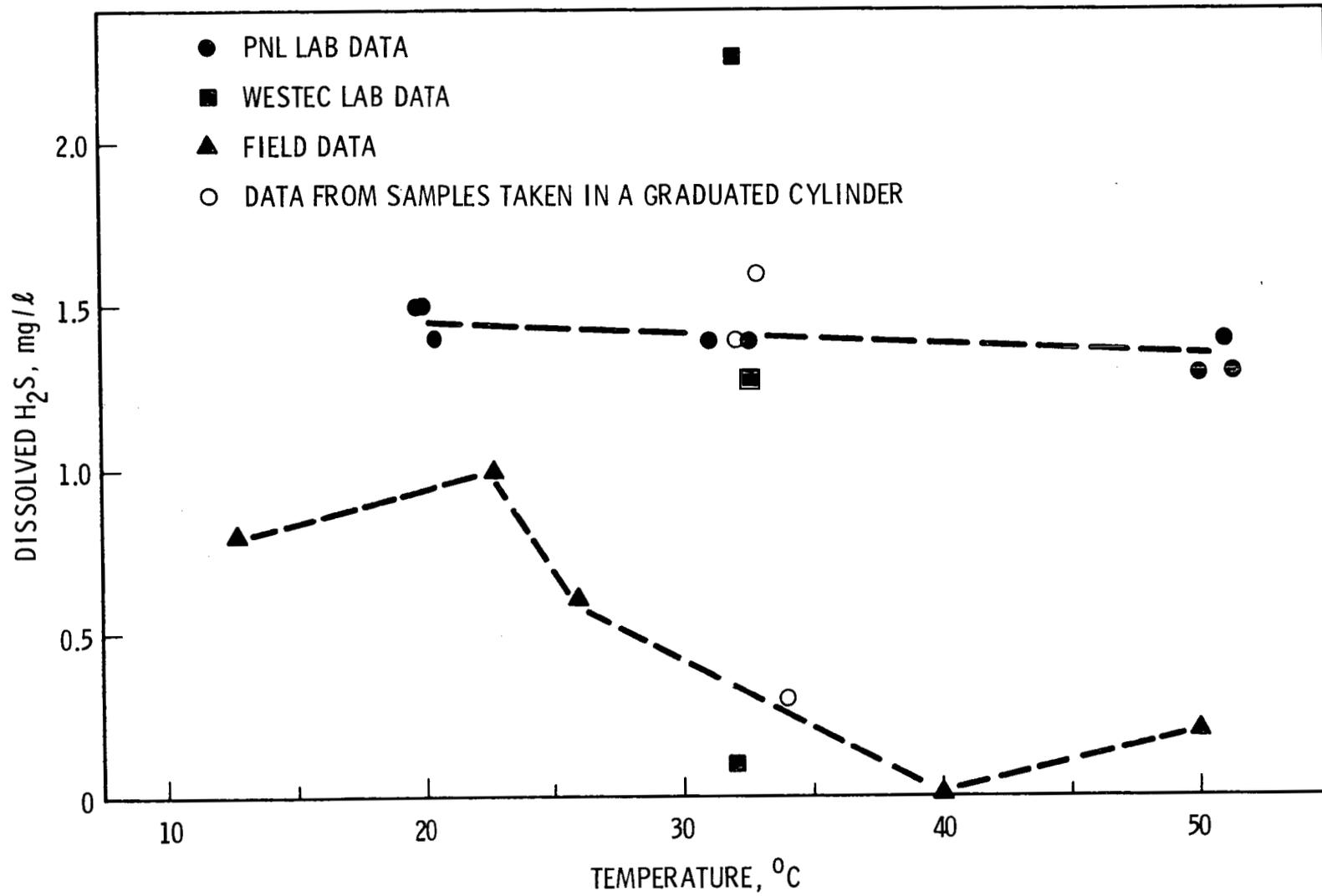


FIGURE 7.1. Variation of Measured H<sub>2</sub>S Content With Temperature

TABLE 7.1. Field and Laboratory Determination of Dissolved H<sub>2</sub>S

Nominal Sampling Temperature, °C	Measured H <sub>2</sub> S mg/l/Measured Sample Temperature, °C		Field Results Colormetric
	Laboratory Results		
	PNL Absorption/Titration Method	Westec Absorption/Titration Method	
10			0.8
20	1.5 1.4		1.0
30 - Sparge in Sample Bottle	1.4 1.4 1.4	2.26 1.28	0.6
Sparge in Graduated Cylinder	1.4 0.3(a) 1.6		
40	--		0
50	1.3 1.3 1.4		

(a) Analyst missed end point.

lab data near 1.5 mg/l are between the 1.0 and 2.0 increments in the high range (1 to 10 mg/l) field comparator. In retrospect, a 2x dilution of the field sample would have produced a useful comparison point, in that it would have resulted in uncorrected readings near the middle of the low range (0 to 1 mg/l) comparator.

It is evident that the field kit should be used as near 20°C as possible. At 25°C and above, the results have begun to fall off rapidly. As temperatures rise above 25°C, gas breakout increases rapidly, tending to reduce the H<sub>2</sub>S content of the liquid. Since only the liquid is analyzed with the field kit, a low H<sub>2</sub>S value results. This deficiency is avoided in the absorption method into H<sub>2</sub>S because here both liquid and gas bubbles are sparged through the Zn acetate.

## 8.0 EVACUATED CYLINDER METHODS (TEST 6)

E. M. Woodruff

The method of attaching an evacuated cylinder to a piping system and valving a sample of the process stream into the cylinder is a common industrial practice. Several difficulties with this method when applied to geothermal systems have been identified:

- The vacuum causes unflashed fluid to flash as it enters the cylinder, thus depositing scale on the inside of the cylinder and lowering the concentration of scale elements in the fluid analysis.
- Metal surfaces exposed to the geothermal fluid can react and enhance the concentration of certain elements.
- The attachment point on the process pipe becomes the sample point, which may introduce contamination, or because it is not in the flowstream may not produce an ideally representative sample.
- Gases and liquids that are combined may not be typical, thereby significantly altering pH, total CO<sub>2</sub> and gas compositions.

To demonstrate some of these effects, this experiment was designed using four cylinders attached at top and side positions at two locations in the loop: behind the first orifice (gas break-out) and behind the second orifice (steam flash conditions).

### 8.1 SAMPLING POINTS

The positions available were Ports 5-31 (side) and 5-33 (top) in the gas break-out leg of the test loop. In the steam flash region, Ports 5-57 (side) and 5-59 (top) were used. Ball valves were attached to the weld-a-lets at the sample ports to shut off flow for sample cylinder attachment and detachment. The cylinders installed on Ports 5-57 and 59 are shown in Figure 8.1. Double shut-off valves were used in series at each end of the 1 l sample cylinders to reduce leak problems. A Tee with a third valve for purging the connecting line was added between the double valves and the ball valves.

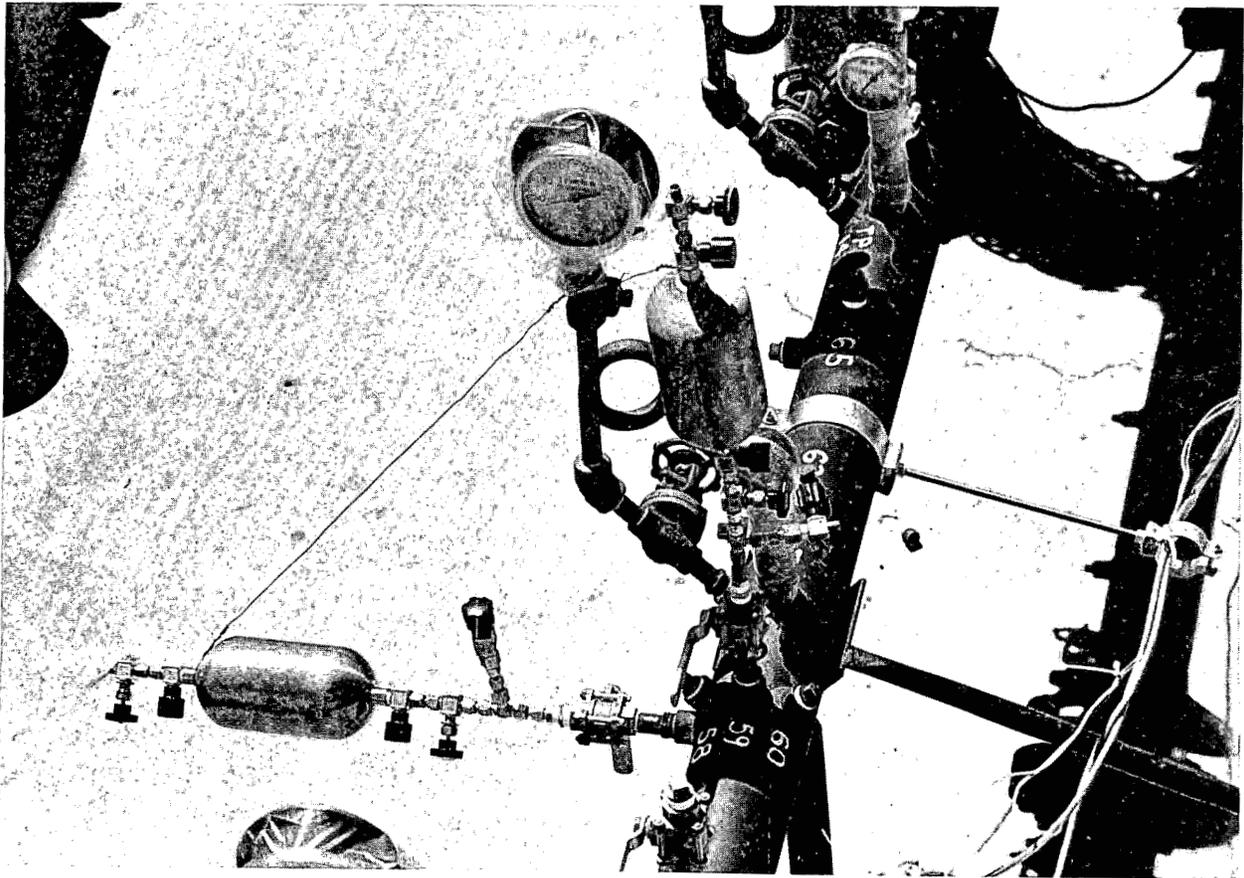


FIGURE 8.1. Evacuated Cylinders - Top and Side Port Attachment to the Test Loop

## 8.2 SAMPLING PROCEDURE

The sequence for sampling was to open the ball valve, open the purge valve and vent for a minute, close the purge valve and open the adjacent pair of bottle valves. After a minute, this procedure was reversed and the cylinders removed with both double valve closures intact.

## 8.3 RESULTS

A number of difficulties prevented assembly of a complete set of data. The data that could be collected (see Table 8.1) reflect limitations in sampling with evacuated cylinders. Data on samples collected with separators and cooling coils are included in the table to allow for inter-method comparison.

TABLE 8.1. Composition of Samples Collected in Steel Cylinders and by Cooling Coils and Separator

Loop Location Sampling Method	Break-Out Region of Gas			Region of Steam Flashing		
	Steel Cylinder		Cooling Coils on Unflashed Brine	Steel Cylinder		Brine (From Weber Separator) Through Cooling Coils
	Side of Loop	Top of Loop	Just Upstream	Side of Loop	Top of Loop	Top of Loop
Analytic Method			B	F-57-01	F-59-01	JB-56-26
pH (Lab)	6.95	No Fluid	5.95	Only 6 ml	6.15	(Field) 8.5
Conductivity	6,600	Recovered	6,790	Fluid Recov-	6,360	--
TDS	4,266		4,240	ered, All	4,123	--
HCO <sub>3</sub>	584		575	Used For CO <sub>2</sub>	1,012	--
CO <sub>2</sub>	1,237		1,155	973	1,867	370
Gas Analysis (mole %)						JB-56-27
CO <sub>2</sub>	4.00	82.30	90.0	81.56	67.64	94.22
Ar <sup>2</sup>	1.15	0.04	0.06	0.08	0.07	0.04
O <sub>2</sub>	1.03	0.04	0.01	0.14	7.18	0.05
N <sub>2</sub>	92.49	2.13	2.56	4.76	6.45	1.34
CO	0.02	<0.1	<0.1	<0.1	0.14	--
He	<0.01	<0.01	<0.01	<0.01	<0.01	--
H <sub>2</sub>	0.22	0.56	0.08	0.03	3.95	0.05
CH <sub>4</sub>	1.01	14.72	7.09	13.26	12.98	4.14
C <sub>2</sub> H <sub>6</sub>	0.08	0.21	0.19	0.17	1.59	0.16
Plasma Results (mg/l)						JB-56-26
Al	0.3		--		0.5	--
As	0.7		0.7		0.7	0.5
B	6.7	Insufficient	7.3	Insufficient	6.8	7.3
Ca	6.3	Sample	9.3	Sample	3.5	2.3
Fe	42.3		0.1		75.5	0.2
K (AA)	121		100		118	--
Li	5.4		5.2		4.9	5.2
Na	1,536		1,374		1,430	1,550
Si	20.5		102		69.3	100
Sr	1.5		2.1		1.3	0.5
Mn	0.4		0.1		0.6	<.05
Ni	83.2		0.1		39.5	<.05

The first pair of steel sample cylinders from the gas break-out region of the loop illustrate the difficulty in obtaining representative samples. In this region of gas break-out, bubbles commonly rise to the top of the pipe and become trapped if there is a restriction to gas flow at some point downstream. In the test loop, the next orifice creates this restriction, and a gas cap forms. Thus, the cylinder connected to the top of the loop collected only gas. The cylinder on the side port collected fluid but leaked; its gas content, therefore, cannot reliably be compared with the companion sample.

In the steam flash region, very little fluid (6 ml) was recovered from the side sample, making fluid analysis impossible. Sufficient fluid was collected from the top for analysis, however, which suggests that turbulence associated with steam flashing at the orifice is great enough to prevent formation of a gas cap. The total dissolved solids content is also high enough to indicate that the steam and brine were well mixed. Our data do not explain the low liquid recovery from the side port nor offer much encouragement that any given sample will be representative.

Fluid reaction within the steel sample cylinders is evidenced by the high in-cylinder Fe and Ni levels compared with samples from separator/cooling coils. Air leaks evidenced in the gas analyses for evacuated cylinder tests may have aggravated cylinder corrosion. Both sample cylinders and cooling coils are made of 304 stainless steel. On the other hand, exposure time in cooling coils, lasting a few seconds, is insignificant even when compared with exposure to well casing and surface piping.

The depletion of fluid constituents resulting from flashing into the evacuated cylinder is shown by reductions in strontium compared with inlet brine. A much larger depletion of silica in steel cylinders (20.5 mg/l versus 102 mg/l from cooling coils) cannot be related to flashing at these concentrations. However, compared with a polyethylene bottle, the affinity of steel surfaces for deposition during transit may account for the factor of 5 difference.

Both gas and liquid are drawn into the cylinder in the steam flashing region. The gas is mostly CO<sub>2</sub>, which redissolves in the liquid or cools,

giving 1) low pH values (6.15 versus 8.5 for separated liquid); 2) too high a CO<sub>2</sub> value (1867 versus 370); and 3) a gas sample depletion in CO<sub>2</sub>, which enriches the other gaseous components. A fourth experimental effect is sample contaminations, which produces high iron and nickel values for the stainless steel cylinder.

#### 8.4 CONCLUSIONS

Convincing arguments have been established against the use of evacuated steel cylinders for collecting geothermal fluid samples destined for comprehensive analyses. The convenience factor of using evacuated cylinder methodology, however, cannot be denied. As long as the limitations of the method are recognized, evacuated cylinder sampling may be applied for collection of single-phase liquid or steam specimens. No further testing is recommended unless specific needs can be identified.

## 9.0 THE MEASUREMENT OF SUSPENDED SOLIDS (TEST 7)

R. P. Smith

### 9.1 INTRODUCTION

Measuring the quantity, size, and distribution of suspended solids in geothermal water is important because precipitates may cause well or formation plugging. Monitoring of suspended solids may also aid in understanding scale formation by indicating whether the scale just grew on the surfaces or in the brine formed as a particulate, which then adhered to the surface. Filter plugging tests have been successfully used by the oil industry to estimate injector-well life span. Despite the importance of suspended solid measurements, a careful study comparing the different techniques has not yet been performed.

During the East Mesa sampling field test, three methods were used to determine suspended solid concentration: filtration, counting with a laser particle counter, and turbidity measurements. These methods were tested on the same day under identical conditions (i.e., same well and sample port, and similar operating procedures). Samples were collected on two days: the first day from Port S-3, which taps single-phase brine, and the second day from Port S-56, which is downstream of the second orifice, which produces a two-phase steam/liquid flow. Thus, more than one kind of flow was tested. Since all three methods to determine suspended solids were collected under identical conditions, variations in the determined amounts should reflect only the accuracy or precision of each method and not sampling or well variation.

This chapter discusses each method separately, then compares the data. The sequence of samples taken by each method is given in Table 9.1.

### 9.2 SUSPENDED SOLIDS DETERMINED BY FILTRATION

Suspended solids were determined by using the standard filter-weight-gain technique with one or a chain of several filters. With the filter chain, suspended-solid samples were collected under in-line temperature and pressure

**TABLE 9.1. Samples Taken For Suspended Solid Measurements, Sampling Conditions**

Sample Method	Location	Sampling Conditions	Volume of Brine (%)	Filter Pore Size (µm)	Sample or Filter No.	Time Taken
Cold Sampling, Single Filter	Port S-3	30°C, 130 psig (inlet)	5	0.45	7-1	8/17/79, 7:18 am
Cold Sampling, Single Filter	Port S-3	30°C, 130 psig (inlet)	5	0.45	7-2	
Cold Sampling, Single Filter	Port S-3	30°C, 130 psig (inlet)	5	0.45	7-3	
Cold Sampling, Filter Chain	Port S-3	27°C, 100-90 psig	5	0.22	7-4	8/17/79 9:30 am
				1.2	7-5	
				5.0	7-6	
				8.0	7-7	
Cold Sampling, Filter Chain	Port S-3	21°C, 70-18 psig	5	8.0	7-8	8/17-79, 10:40 am
				5.0	7-9	
				1.2	7-10	
				0.22	7-11	
Laser Counting	Port S-3		0.2	1.2	7-12, B-07	8/17/79, 11:10 am
Filter Samples			0.2	5.0	7-13, B-08	
			0.2	8.0	7-14, B-09	
Raw Brine			0.2	--	B-01	
			0.2		B-02	
			0.2		B-03	
Acidified Brine			0.2		B-04	
			0.2		B-05	
			0.2		B-06	
Cold Sampling, Filter Chain	Port S-3	35°C, 85-78 psig	5	8.0	7-15	8/17/79, 1:20 pm
				5.0	7-16	
				1.2	7-17	
				0.22	7-18	
Hot Sampling Filter Chain	Port S-3	156°C, 47-38 psig	5	10.0	7-19	8/17/79, 4:20 pm
				5.0	7-20	
				3.0	7-21	
				1.0	7-22	
				0.2	7-23	
Hot Sampling, Filter Chain	Port S-3	160°C, 115-100 psig	5.15	10.0	7-24	8/17/79, 5:15 pm
				5.0	7-25	
				3.0	7-26	
				1.0	7-27	
				0.2	7-28	
Hot Sampling, Filter Chain	Port S-3	135°C, 110-100 psig	5	10.0	7-29	8/17/79, 6:00 pm
				5.0	7-30	
				3.0	7-31	
				1.0	7-32	
				0.2	7-33	
Cold Sampling, Single Filter	Port S-56	30°C, 70 psig	5.25	0.45	7-34	8/18/79, 9:03 am
Cold Sampling, Single Filter	Port S-56	30°C, 70 psig	5.1	0.45	7-35	8/18/79, 9:22 am
Cold Sampling, Filter Chain	Port S-56	32°C, 78-1 psig	5.14	8.0	7-36	8/18/79, 10:00 am
				5.0	7-37	
				1.2	7-38	
				0.45	7-39	
				0.22	7-40	
Cold Sampling, Filter Chain	Port S-56	35°C, 78-1 psig	5	8.0	7-41	8/18/79, 10:30 am
				5.0	7-42	
				1.2	7-43	
				0.45	7-44	
				0.22	7-45	
Turbidity	Port S-56				T-1	8/18/79, 11:09 am
					T-2	
					T-3	
Laser Counter	Port S-56		0.2		B-10	8/18/79, 11:10 am
Raw Brine			0.2		B-11	
			0.2		B-12	
Turbidity	Port S-3				T-4	8/18/79, 11:17 am
					T-5	
					T-6	

conditions, and in a cooled (20°C) sample, which represents the most typical method used. The data for these two sampling conditions will be compared to determine if, in cooling the brine, the quantity and makeup of the suspended solids are changed. In all cases, an insertion probe was used to collect the brine from the center of the pipe. (We sampled from the center in order to avoid contamination by solids that may have settled on the pipe wall.) Figure 9.1 details the sampling system for both hot and cold sampling, while Figure 9.2 provides a more comprehensive picture of the filter chain suspended-solid sampler.

#### 9.2.1 Procedure

Procedures for all of the filtration tests were similar. Filters were placed in an oven at 60°C for 2 hr to remove any moisture, were cooled, and then weighed. We checked several filters to see if all the water had been removed from them during this initial drying. The reheating and reweighing produced stable weights, which implies that all moisture was removed. After a sample had been collected, the filters were again dried at 60°C for 2 hr and reweighed. The step-by-step procedure for sampling is listed below:

1. Fill suspended-solid sampler with DI water to prevent flashing when the inlet valve is first opened. Attach suspended-solid sampler to sample port, all valves closed.
2. Slowly open the inlet valve to pressurize system.
3. Slowly open the exit valve, then the control valve to initiate flow.
4. By controlling flow with the control valve, keep the pressure drop across the supported filter membranes within structure limits and above flashing.
5. When the flow drops slightly (indicating filter plugging) or when the predetermined amount of brine has passed through the filters, close valves starting from the control valve and go upstream to the ball valves.
6. Record total brine flow, brine temperature, pressure data, and sample port data.
7. Carefully remove filters, replace all or only the particular filter(s) that plugged up.

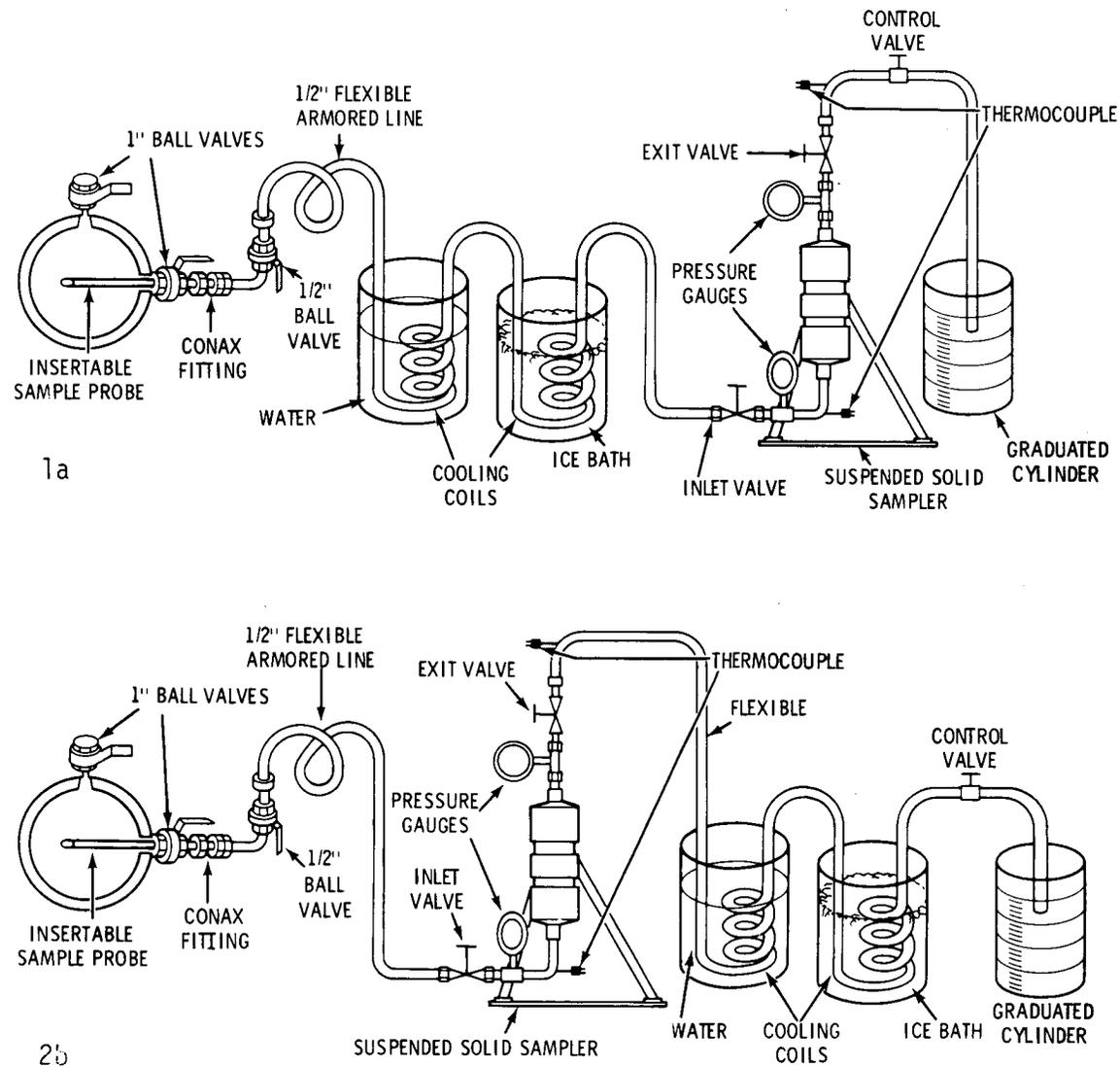


FIGURE 9.1. Sampling System For Collecting and Measuring Suspended Solids; (a) System For Cold Sampling, (b) System For Hot Sampling

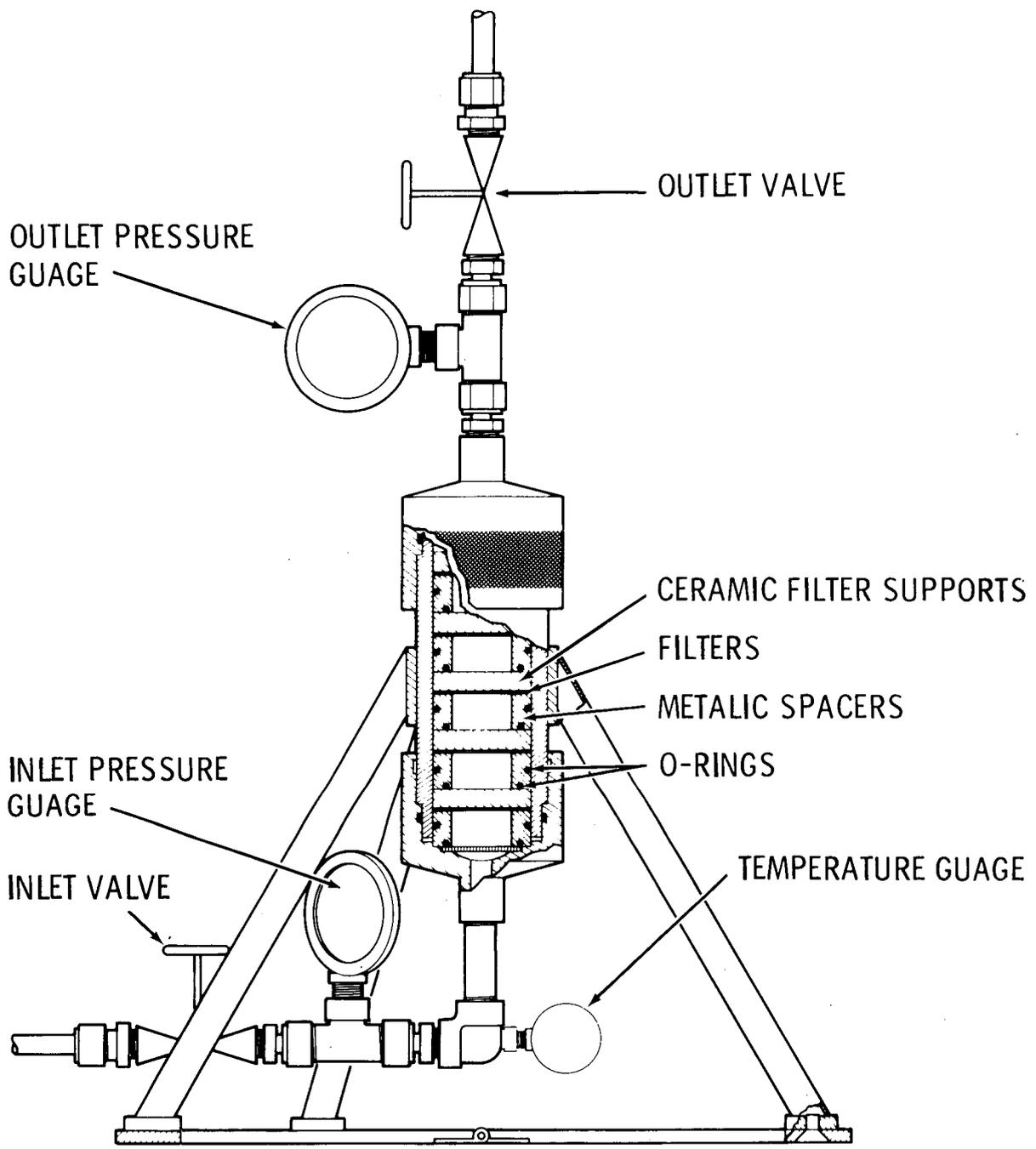


FIGURE 9.2. Cross-Section of Suspended Solid Sampler Showing The Location of the Filter Supports and Gauges

### 9.2.2 Results of Filtration Tests

Using a single filter (0.45  $\mu$  pore diameter) the total suspended solids were determined to be 0.26 mg/l at Port S-3 (single-phase flow) and 0.77 mg/l at Port S-56 (two-phase flow). From this data, it appears that the quantity of suspended solids increases after the brine flashes. Suspended solids were also collected in a filter chain to determine the size distribution of these solids and to collect them for later chemical analysis. Again, the two-phase regime contained more total suspended solids: 1.83 mg/l versus 1.15 mg/l. Table 9.2 lists the results of determining suspended solids by cold filtration. It is interesting to note that the increase of mg/l for two-phase versus single-phase sampling is mostly attributable to the capacity of the 0.45 and 0.22  $\mu$  filters to trap the smaller particles. A sample calculation done for Filter 7-2, which shows background and salt corrections, is presented in Appendix A.

More solids were collected by the filter-chain technique than by the single-filter technique. The additional weight can partially be attributed to brine residue on the filters, which was counted into the total weight of solids either four or five times instead of just once. This was verified by XRF analysis, in which the chloride was found to be evenly distributed among the various filter sizes. The quantity of chloride in  $\mu\text{g}/\text{cm}^2$  was then multiplied by 13.85  $\text{cm}^2$ , the area of the filter; then by 1.65 to convert from  $\mu\text{g}$  Cl to  $\mu\text{g}$  NaCl. This quantity of salt was then subtracted from the filter weights. This method, though somewhat awkward compared to rinsing the filters with distilled water, does assure that none of the sample has been washed away. It should be emphasized that the filters must be rinsed or salt correction added. Now after experience in both methods, rinsing the filters in their holders is the most convenient method.

Even after rinse correcting for dried salt residue, however, the filter-chain method still produced higher suspended solid concentrations than the single-filter technique (see Table 9.11). This can be partially attributed to actually collecting more solids using a smaller pore size (0.22  $\mu$  vs. 0.45  $\mu$ ) filter. Otherwise, the difference in collection capacity may mean that a filter chain is a more effective way of collecting suspended solids.

TABLE 9.2. Total Suspended Solids by Filtration of Cooled Brine

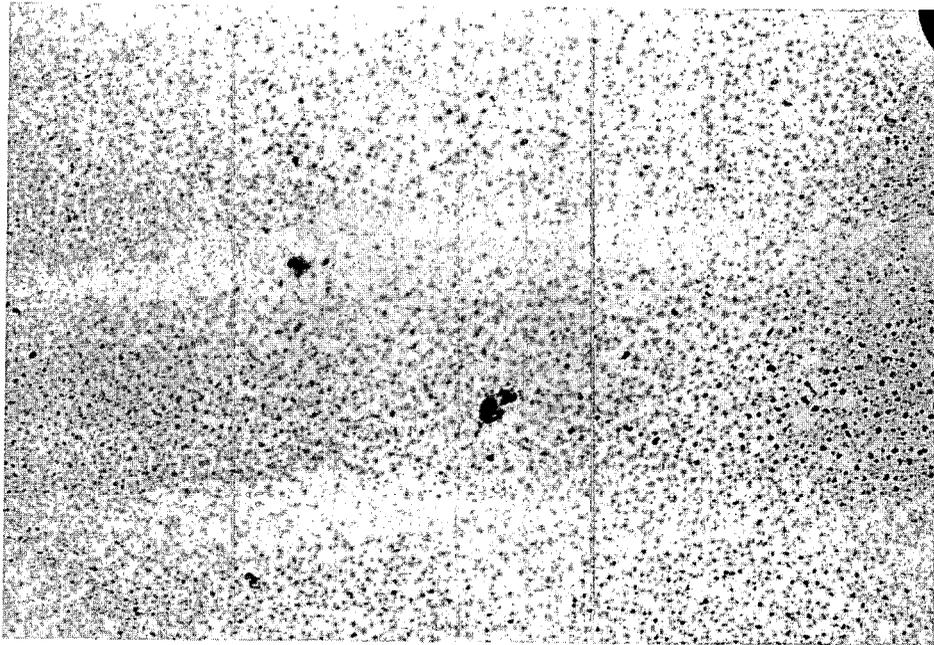
	Port S-3 (Single-Phase)		Port S-56 (Two-Phase Flow)	
	Filter No.	Weight Gain	Filter No.	Weight Gain
Total Suspended Solids (0.45 $\mu$ Filter)	7-1	0.0017 g/5 l	7-34	0.0043 g/5.25 l
	7-2	0.0017	7-35(a)	0.0018 g/5.1 l
	7-3(a)	0.0013		
	Ave	1.7 mg/5 l	Ave	4.3 g/5.25 l
	(- Salt)	<u>0.38</u>	(- Salt)	<u>0.28</u>
TOTAL		0.26 mg/l		0.77 mg/l
Suspended Solids Sized Distribution Filter Chain	7-7 (8.0 $\mu$ )	1.6	7-36 (8.0 $\mu$ )	2.1
	7-8	2.3	7-41	<u>1.6</u>
	7-15	<u>1.6</u>		
	TOTAL	1.8 Ave		1.9
	7-6 (5.0 $\mu$ )	2.2	7-37 (5.0 $\mu$ )	1.7
	7-9	1.6	7-42	<u>2.1</u>
	7-16	<u>2.0</u>		<u>1.9</u>
	TOTAL	1.9 Ave		
	7-5 (1.2 $\mu$ )	2.2	7-38 (1.2 $\mu$ )	2.0
	7-10	1.7	7-43	<u>1.9</u>
	7-17	<u>1.6</u>		
	TOTAL	1.8 Ave		2.0
			7-39(a) (0.45 $\mu$ )	1.5
			7-44	<u>3.7</u>
				<u>3.7</u>
	7-4 (0.22 $\mu$ )(b)	4.8	7-40 (0.22 $\mu$ )	2.5
	7-11	1.8	7-45	<u>4.1</u>
	7-18	<u>1.6</u>		
	TOTAL	1.7 Ave		3.3
TOTAL	Total (-4X Salt)	7.2 mg/5 l	Total (-5X Salt)	12.8 mg/5.25 l
		1.44		3.2
		5.76 mg/5 l		9.6 mg/5.25 l
TOTAL		<u>1.15 mg/l</u>		<u>1.83 mg/l</u>

(a) Developed small holes.  
(b) Filter broke.

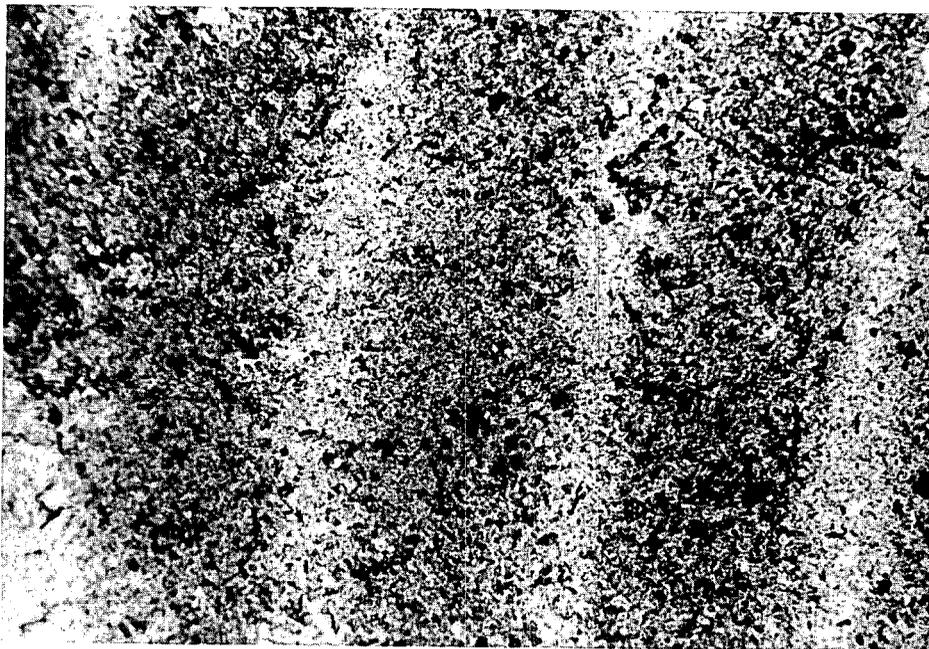
### 9.2.3 Composition of Residue on Filters

The suspended solids collected on the filters were analyzed using x-ray fluorescence and x-ray diffraction techniques, and optical microscope observations. This is the first complete study of different-size fractions of suspended solids in a geothermal system. Some x-ray studies have been done of two sizes of larger suspended solids at Magnamax Well #1 (Hill and Ott, 1977). Under x-ray diffraction, no crystalline phases were detected. The amorphous state of the suspended solids was varified by observations under polarized light with filters treated to make them transparent to light. The filter clearing procedure we used is credited in Reference 9.2. Except for a scattering of quartz grains, all the solids on the filters were amorphous. Figure 9.3 shows photomicrographs displaying the difference in quantity of suspended solids between the one- and two-phase flows. The results of the x-ray fluorecence analysis are reported in Table 9.3.

Major trends were manifested for Si, S, Fe, and Ca among the various-sized filters. These trends are clearly seen in Table 9.4 where the blank has been subtracted. At Port S-3 (single-phase), Ca is more concentrated in the large-pore filters; S and Fe were collected on the finer-sized filters; Si was most concentrated on the finer-sized and larger filters; and Cl was evenly distributed among the various-sized filters. At Port S-56 (two-phase flow), most of the elements, except Ca, appear to have a bimodel distribution. Again, Ca is mostly trapped on the larger-sized filters. For all three sample modes (Port S-3, hot and cold, and Port S-56), the concentration of Fe and S favored similar-sized fractions, which suggests that these elements are chemically bonded. In fact, the yellow-orange color dominate on the filters was most likely produced by this iron-sulfide coalescence. Samples collected at Port S-56 following the brine flash did not contain increased amounts of Ca. Aragonite scale was being deposited on the pipe at this sample port, yet suspended particulates of aragonite ( $\text{CaCO}_3$ ) were not observed. This phenomenon suggests that scale crystals grow by ion attraction onto the crystal lattice on the pipe walls, and are not formed by nucleation and then adherence to the pipe wall.



PORT S-3  
FILTER #7-2



PORT S-56  
FILTER #7-34

0.14 cm

FIGURE 9.3. Photomicrographs of Total Suspended Solids at Ports S-3 and S-56 (Filter Size is  $0.45 \mu$ )

TABLE 9.3. X-Ray Fluorescence Analysis of Suspended Solids

Sample	Si	P	S	Cl	K	Ca	Li	V	Cr	Mn	Fe	Ni	Cu	Zn	Ga	Hg	Se	Pb	As	Br	Rb	Sr	Minerals
1. LD Blank	<7.2	<2.9	<1.3	.59 +.29	<.16	<.11	<.04	<.03	.02 +.006	<.01	.027 +.008	.008	.026 +.005	.015 +.004	<.006	.024	<.007	<.03	<.005	.007	<.010	<.083	--
2. 7-20	85.5 +20.6	<6.3	5.1 +2.0	14.4 +1.5	1.14 +.26	2.1 +.23	.11 +.05	<.05	.62 +.60	.31 +.03	13.0 +.92	.39 +.03	.11 +.01	.805 +.060	<.009	.31 +.03	<.008	.19 +.02	.050 +.009	.28 +.02	<.014	<.090	--
3. 7-19	431.3 +37.1	<8.5	8.3 +2.5	6.5 +1.2	.78 +.21	1.2 +.18	.73 +.09	<.08	4.8 +.34	20.2 +1.4	23.9 +1.7	<.006	.32 +.03	.407 +.032	<.010	.16 +.03	<.010	.21 +.03	.039 +.009	.067 +.008	<.014	<.011	--
4. FG Blank	<2.9	<1.4	<.57	<.29	<.09	<.05	.05 +.02	<.02	.02	<.01	.012 +.005	7.3 +.52	.020 +.004	<.005	<.005	.019	<.006	.02	<.004	<.005	<.009	<.069	--
5. 7-32	14.5 +2.1	<1.1	.97 +.50	4.6 +.50	.33 +.08	.41 +.06	.55 +.06	<.03	.03 +.01	.012 +.006	1.26 +.10	.030 +.006	.032 +.005	.150 +.014	<.004	.088 +.015	<.004	.02	.016 +.004	.067 +.007	<.008	<.053	--
6. 7-33	6.2 +2.3	<1.6	<.79	4.6 +.53	.42 +.09	.14 +.05	.23 +.04	<.03	.07 +.01	.259 +.080	1.02 +.08	.067 +.009	.021 +.005	.186 +.016	<.005	.020	<.006	.02	<.005	.085 +.008	<.010	<.070	--
7. FS Blank	<14.0	<1.3	3.5 +.52	.27	.26 +.07	.12 +.04	.06 +.02	<.02	.01	<.01	.024 +.006	<.005	.013 +.004	.010 +.003	<.005	.018	<.006	.02	<.004	<.006	<.009	.65 +.06	--
8. 7-31	<17.1	<1.9	3.8 +.86	9.01 +.83	.76 +.07	.97 +.11	.12 +.03	<.03	.07 +.01	.020 +.003	1.18 +.09	.052 +.007	.042 +.007	.181 +.16	<.006	.22 +.03	<.006	.03	<.010	.059 +.007	<.010	.63 +.06	--
9. SC Blank	<1.9	<.90	.46 +.17	.46	.08 +.04	.20 +.05	<.02	<.02	.01	<.01	.025 +.006	<.004	.019 +.004	.022 +.004	<.004	.014	<.004	.02	<.008	.010 +.003	<.007	<.056	--
10. 7-8	<11.7	<1.5	1.3 +.64	15.03 +1.28	1.43 +.16	.76 +.11	.10 +.03	<.02	.11 +.02	.040 +.001	.70 +.06	.158 +.015	.051 +.007	.180 +.016	<.005	.10 +.02	<.004	.03	.056 +.007	.028 +.005	<.008	<.061	--
11. 7-36	18.8 +7.0	<2.0	2.8 +.97	42.01 +3.14	282 +.26	.90 +.13	<.06	<.03	2.4 +.18	.47 +.04	11.4 +.81	1.02 +.076	.037 +.009	.103 +.010	<.005	.037	<.005	.08 +.01	.053 +.009	.045 +.006	.013 +.005	<.065	Siderite
12. 7-37	3.7 +1.2	.96	.88 +.37	13.46 +1.1	1.22 +.14	.40 +.07	<.03	<.02	.18 +.02	.04 +.01	.89 +.07	.083 +.010	.020 +.004	.068 +.008	<.004	.021	<.004	.02	.012 +.003	.016 +.004	.008 +.004	<.052	--
13. 7-9	<10.3	<1.4	2.0 +.67	20.17 +1.59	1.43 +.17	.43 +.90	.16 +.03	<.02	.05 +.01	.044 +.008	.44 +.04	.17 +.02	.036 +.006	.288 +.023	<.005	.11 +.02	<.004	.04	.082 +.009	.044 +.006	.016 +.004	<.054	Siderite
14. 7-10	3.1 +1.5	<1.4	<1.2	18.69 +1.49	1.75 +.18	.42 +.07	<.03	<.02	.03 +.01	<.01	.17 +.02	.22 +.02	.049 +.007	.104 +.010	<.005	.02	<.005	.02	.014 +.004	.021 +.004	<.008	<.059	--
15. 7-38	5.9 +1.7	<1.5	1.4 +.65	15.76 +1.29	1.30 +.15	.22 +.07	<.03	<.02	.22 +.03	.655 +.001	2.14 +.16	.16 +.01	.045 +.007	.044 +.006	<.004	.04 +.01	<.005	<.02	.015 +.005	.029 +.004	<.008	<.061	--
16. 7-11	22.7 +8.9	<2.5	8.9 +1.3	12.8 +1.2	1.6 +.25	.36 +.11	.14 +.03	<.02	.092 +.016	.025 +.009	1.08 +.08	.44 +.04	.060 +.009	1.3 +.09	<.008	.33 +.04	<.006	<.03	.33 +.03	.063 +.008	<.010	<.071	--
17. 7-40	16.0 +3.2	<2.1	<1.9	46.2 +3.5	3.0 +.28	.38 +.08	<.04	<.02	.078 +.015	.020 +.009	2.9 +.21	.13 +.01	.028 +.006	.076 +.009	<.005	.03	<.006	<.02	.058 +.007	.059 +.006	<.010	<.069	--
18. Gg Blank	4.1 +1.7	<1.5	.86 +.34	.46 +.23	.12 +.06	.28 +.06	<.03	<.02	<.017	<.012	.034 +.007	<.006	.025 +.005	.030 +.005	<.005	.02	<.006	<.02	<.005	.021 +.004	<.009	<.070	--
19. Test 1	16.8 +3.0	<2.5	9.3 +1.4	15.9 +1.4	1.3 +.19	<.46	<.08	<.02	.050 +.014	<.016	1.37 +.10	.24 +.02	.060 +.009	.25 +.02	<.007	.43 +.05	<.006	<.03	.08 +.01	.058 +.009	<.014	<.069	--
20. 7-2	<4.5	<1.9	4.2 +.85	17.1 +1.4	1.5 +.19	<.35	<.06	<.02	.028 +.010	<.013	.59 +.05	.021 +.005	.043 +.007	.073 +.008	<.005	.21 +.04	<.006	<.03	<.014	.048 +.007	<.010	<.069	--
21. 7-34	59.7 +18.3	<3.7	16.0 +1.8	12.5 +1.2	2.3 +.35	20.4 +1.5	.17 +.05	<.05	1.28 +.10	.360 +.037	29.6 +2.1	1.5 +.11	.099 +.014	.41 +.03	<.007	<.07	<.007	<.03	.57 +.04	.116 +.012	<.011	3.6 +.26	--
22. 7-39	26.1 +8.6	<2.3	2.3 +9.1	28.1 +2.2	2.1 +.21	.65 +.10	<.06	<.03	.43 +.04	.114 +.017	10.4 +.74	.32 +.03	.032 +.007	.12 +.01	<.006	<.03	<.005	.06 +.01	.031 +.01	.031 +.005	<.010	<.069	Magnetite

9.10

TABLE 9.4. Element Versus Size Distribution

	Filter No.	Pore Size $\mu$	Element Concentration in $\mu\text{g}/\text{cm}^2$				
			Si	S	Cl	Ca	Fe
Port S-3	7-11	0.22	20.8	8.44	12.34	0.16	1.06
	7-10	1.2	1.2	3.74	18.23	0.22	0.15
	7-9	5.0	8.4	1.54	10.71	0.23	0.42
	7-8	8.0	9.8	0.84	14.54	0.56	0.68
Port S-56	7-40	0.22	14.1	4.44	45.74	0.18	2.88
	7-39	0.45	22.0	1.84	27.74	0.45	10.38
	7-38	1.2	4.0	0.94	15.30	0.02	2.12
	7-37	5.0	1.8	0.42	10.00	0.20	0.87
	7-36	8.0	16.9	2.34	41.55	0.70	11.38
Port S-3 (hot)	7-33	0.2	3.3	ND	4.6	0.09	1.01
	7-32	1.0	11.6	ND	4.6	0.36	1.25
	7-31	3.0	3.1	0.3	9.0	0.85	1.16
	7-20	5.0	78.3	0.8	13.8	1.99	12.97
	7-19	10.0	424.1	4.0	5.9	1.09	23.87

ND = Not Detected

#### 9.2.4 Comparison of Cold and Hot Filter Sampling

This section compares the cold filter sampling method with the hot sampling method. Table 9.5 presents the results of collecting suspended solids under the in-line temperature of 163°C. More suspended solids (8.5 mg/l) are collected under hot conditions than under cooled, depressurized conditions (1.15 mg/l). This finding is important because it casts doubt on the standard method currently used to obtain a suspended solid sample, which involves cooling the brine to 20°C before flushing it through a filter. During the first hot sampling test, the pressure in the suspended solids sampler was not kept high enough to prevent the water from flashing. During the second and third sampling, however, there was sufficient pressure to prevent flashing. Even for these cases, there was still a greater weight gain of solids collected during hot sampling than during cold sampling.

Whereas the suspended solids were evenly distributed among the four size fractions during the cold sampling, the larger size fractions (10.0 and 5.0  $\mu$ )

TABLE 9.5. Total Suspended Solids By Filtration Using Hot Sampling Techniques, Port S-3

<u>Filter No.</u>	<u>Filter Pore Size</u>	<u>mg/5 Liters of Brine</u>	<u>Highest T With Lowest P During Sampling</u>	<u>Vapor Pressure of Water at Sampling Temperature</u>
7-19	10.0 $\mu$	46.2 <sup>(a)</sup>		
7-24		8.4 <sup>(b)</sup>		
7-29		25.3		
		<u>25.3 Ave</u>		
7-20	5.0 $\mu$	34.9 <sup>(a)</sup>		
7-25		8.6		
7-30		5.2		
		<u>6.9 Ave</u>		
7-21	3.0 $\mu$	5.0 <sup>(a)</sup>		
7-26		3.2		
7-31		3.4		
		<u>3.3 Ave</u>		
7-22	1.0 $\mu$	3.9 <sup>(a)</sup>		
7-27		4.7		
7-32		2.5		
		<u>3.6 Ave</u>		
7-23	0.2 $\mu$	4.5 <sup>(a)</sup>		
7-28		4.9		
7-33		3.2		
		<u>4.1 Ave</u>		
	Total	43.2 mg/5 l		
	-(5X Salt)	0.8		
	Correction	<u>42.4</u>		
		<u>8.48 mg/l</u>		
7-19 $\rightarrow$ 7-23			163°C, 38 psig	82 psig
7-24 $\rightarrow$ 7-28			160°C, 90 psig	75 psig
7-29 $\rightarrow$ 7-33			124°C, 97 psig	18 psig

(a) Brine flashed, was not included in average.

(b) Small holes developed, was not included in average.

contained a disproportionate amount of the suspended solids collected during hot sampling. Also, these sized fractions contained substantial amounts of Si. The samples will be further studied by electron microscopy. If quartz grains are responsible for the additional weight, it would follow that the larger particles tend to settle in the cooling coils during cold sampling.

### 9.3 SUSPENDED SOLIDS DETERMINED BY LASER PARTICLE COUNTING

The quantity of suspended solids of varying diameter was determined using a Prototon Particle Counter®, Model ILI 1000 plus the Particle Profile Attachment. Samples were collected in 8 oz. clear glass bottles that were previously rinsed with filtered (0.45  $\mu$ ) distilled water. The brine was cooled and depressurized through the sampling system used for the filtered samples (Figure 9.1).

#### 9.3.1 Procedures

The procedure for counting is listed below:

1. Check to see that the laser particle counter is operating properly by testing standard suspension solutions. (Slight modifications can be made by adjusting the distances between the lenses.)
2. Determine the background counts for each specific field location by setting the threshold setting at a value low enough to get 10 counts in the scan period for a blank solution. The instrument may need to be sheltered from bright light.
3. Set threshold limit using calibration chart after determining background level and the particle size diameter of the smallest particle of interest.
4. Gently agitate the sample of bottled liquid to produce a uniform suspension.
5. Place bottle in the "V" notch and rotate to a point where the laser beam enters and leaves unobstructed.

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6. Press the "count" button two minutes after bottle agitation. Within 15 seconds, the total particle count per ml of particles greater than a given diameter is displayed on the digital readout. If the counts are greater than 1,000, increase the threshold limit.
7. When using the Particle Profile Attachment: the quantity of particles of varying diameters in increments of 5  $\mu$  are printed along with a histogram of the size distribution (the threshold setting on the counter does not effect this attachment, thus, 1  $\mu\text{m}$  is the minimum diameter).
8. Count each bottle 10 times and average the counts.

### 9.3.2 Results

Three samples of raw brine were collected at Sample Ports S-3 (B-01, B-02, B-03) and S-56 (B-10, B-11, B-12) for suspended solids determination. Three additional brine samples from Port S-3 (B-04, B-05, B-06) were acidified with five drops concentrated HCl to determine if acidification preserved the samples. Three samples (B-07, B-08, B-09) were prefiltered to discern the effects of removing fine-sized particles on solids growth.

Previous studies by Grens (Ref. 9.3) show that the quantity of suspended solids increases with time. This finding is important because geothermal samples are often collected in the field, then transported back to the laboratory for analysis, sometimes stretching the time between sampling and analysis to several days. To test for this phenomena, we monitored suspended solids concentration in the brine at the time the sample was collected and at various times throughout the samples shelf life, up to 140 hr following collection. Table 9.6 lists when the laser samples were collected and counted.

At Port S-3 (see Table 9.7), the suspended solids totals for the three bottles were 233, 179, and 398 particles per ml. The scatter in the values indicates that three 250 ml bottles of brine do not constitute a statistically representative sample. It is interesting to note that the acidified samples were higher in total suspended solids (485, 458, and 612 particles/ml). Apparently, acidification of this particular brine does not preserve the sample for suspended solid measurements. The increase in total suspended solids is most likely due to accelerated silica precipitation as pH decreases.

TABLE 9.6. Counting Schedule For Laser Samples

<u>Sample Port and Samples</u>	<u>Time and Date of Reading</u>	<u>Time Since Initial Sampling (hr)</u>
Port S-3	11:15 am, 8/17/79	Initial Sampling
B-01, B-02, B-03	12:15 pm, 8/17/79	1.0
B-04, B-05, B-06	6:15 pm, 8/17/79	7.0
B-07, B-08, B-09	6:15 pm, 8/18/79	19.0
	4:30 pm, 8/18/79	19.25
	11:15 am, 8/19/79	48.0
	7:30 am, 8/20/79	68.25
	9:00 am, 8/21/79	94.75
	7:30 am, 8/23/79	140.25
Port S-56	11:15 am, 8/13/79	Initial Sampling
B-10, B-11, B-12	11:20 am, 8/18/79	0
	12:15 pm, 8/18/79	1.0
	4:15 pm, 8/18/79	5.0
	11:15 am, 8/19/79	24.0
	7:30 am, 8/20/79	44.75
	9:00 am, 8/21/79	70.25
	7:30 am, 8/23/79	116.25

The concentration of solids in raw samples (Port S-3) increased during the 95 hr after sampling, then decreased at 140 hr. The decrease may have occurred because the solids obtained sufficient mass to settle out. At Sample Port S-56 (Table 9.8), similar trends were observed. Suspended solids reached their maximum level at 24.0 hr, then decreased. This variation in suspended solids with time proves that suspended solid measurements should be made as soon after sampling as possible.

At Sample Port S-56, the suspended solids totals among the three samples did not vary as much. Counts were 744, 937 and 893 particles per ml, respectively. Comparing the average for the three at S-56 (858) to the average for the three raw samples at Port S-3 (270), it would appear that more suspended solids are contained in the brine after flashing than if flashing does not occur.

TABLE 9.7. Total Suspended Solids Variation With Time From Port S-3 Using the Laser Particle Counter

Sample/hr	1.0	2.0	7.0	19.0	29.25	48.0	68.25	94.75	140.25
B-01, Raw Brine	233	213	177	257	280	338	260	612	485
B-02, Raw Brine	179	123	115	216	394	426	348	707	283
B-03, Raw Brine	398	676	560	506	381	638	296	451	373
B-04, Acidified	485	479	625	774	760	(T = 15) 402	(T = 15) 211	(T = 15) 390	(T = 15) 387
B-05, Acidified	458	416	658	981	933	916	(T = 15) 460	(T = 15) 608	(T = 5) 865
B-06, Acidified	612	797	690	765	962	852	(T = 5) 814	(T = 15) 477	(T = 15) 400

T = Threshold Setting, 3.5  $\mu$  if not otherwise specified

TABLE 9.8. Total Suspended Solids Variation With Time From Port S-56 Using the Laser Particle Counter

Sample/hr	0	1.0	5.0	24.0	44.75	70.25	116.25
B-10, Raw Brine	744	893	893	(T = 7.5) 982	(T = 7.5) 908	(T = 7.5) 722	(T = 7.5) 887
B-11, Raw Brine	937	885	853	951	885	(T = 7.5) 815	(T = 7.5) 775
B-12, Raw Brine	893	(T = 7.0) 905	(T = 7.0) 906	(T = 7.0) 926	(T = 7.0) 894	(T = 7.5) 885	(T = 7.5) 740
Average	858						

T = Threshold Setting; 5.0  $\mu$  unless otherwise specified

The size distribution of the solids was determined using a Particle Profile Attachment to the Laser Particle Counter. Table 9.9 presents size distribution data for Sample B-02 at various times after sampling. The finest-sized particles (1-5  $\mu$ ) make up 83% of the total counts (644), and each successively larger size range makes up less and less of the total counts. The suspended solids size distribution changes slightly with time: the finest size range (1-5  $\mu$ ) becomes slightly less dominant, and the next size range (5-10  $\mu$ ) increases at a faster rate.

TABLE 9.9. Variation in Particle Size Distribution With Time, Sample B-02

Size Range	Counts (Particles/ml) For Each Size Range							
	Hours After Sampling							
	1.0	7.0	19.0	19.25	48.0	68.25	94.75	140.25
1-5 $\mu$	534	608	629	797	680	785	727	778
5-10 $\mu$	72	113	94	224	224	223	569	179
10-15 $\mu$	21	19	21	33	46	66	173	45
15-20 $\mu$	7	5	8	10	36	18	60	18
20-25 $\mu$	4	2	5	4	27	6	27	8
25-30 $\mu$	2	1	2	3	5	4	14	4
30-35 $\mu$	1	0	1	1	3	2	7	3
35-40 $\mu$	0	1	1	1	1	1	5	2
40-45 $\mu$	0	0	0	1	1	1	3	2
45-50 $\mu$	1	0	0	0	1	0	3	1
50-55 $\mu$	1	0	0	0	0	0	2	0
55-60 $\mu$	0	0	0	0	0	0	1	1
60-65 $\mu$	0	0	0	0	0	0	1	0
65-70 $\mu$	0	0	0	0	0	0	2	0
70-75 $\mu$	0	0	0	0	0	0	1	0
75-80 $\mu$	0	0	0	0	0	0	1	0
% of 1-5 $\mu$	83	81	83	74	67	71	46	75
Total Counts	644	752	762	1,074	1,022	1,108	1,597	1,042

#### 9.4 SUSPENDED SOLIDS DETERMINED BY TURBIDITY MEASUREMENTS

Turbidity of the brine in Formazin Turbidity Units (FTU) was also determined simultaneously with the other sampling methods at Ports S-3 and S-56. The turbidity data will be compared to the other methods of suspended solid sampling to determine how accurately it measures suspended solids. Turbidity measurements are relatively quick and easy to perform, and since most laboratories have a spectrophotometer, the cost is also minimal.

#### 9.4.1 Procedure

The procedure used for turbidity measurements is listed below:

1. Measure 25 ml of the brine into a clean sample cell.
2. Fill another sample cell with about 25 ml of clear, colorless water and place it in the cell holder.
3. Insert the Turbidity (Absorptometric Method) Meter Scale in the meter and adjust the wavelength dial to 450 nm. Adjust the light control for a meter reading of zero units.
4. Using turbidity standards, check the calibration of the meter scale.
5. Place the sample in the cell holder and read the Formazin Turbidity Units (FTU) from the scale.

At the East Mesa sampling field test, a Bausch and Lomb Minispec 20 was employed for the determinations. The results are listed in Table 9.10. The smallest values were obtained at the single-phase Sampling Port S-3, again implying that more suspended solids are present after flashing.

TABLE 9.10. Turbidity Measurements

<u>Sample Port</u>	<u>Sample</u>	<u>Turbidity (FTU)</u>
S-3	T-1	1.7 <sup>(a)</sup>
	T-2	0.22
	T-3	0.23
	Average	0.23
S-56	T-4	0.94
	T-5	0.26
	T-6	0.51
	Average	0.57

(a) Sampling system was not fully flushed.

## 9.5 COMPARISON OF THE THREE METHODS TO MEASURE SUSPENDED SOLIDS

Each of the three methods: filtration, laser counting, and turbidity measurements; recognized a higher quantity of suspended solids at Port S-56, which was located downstream of an orifice where flashing occurred. The results from each method of suspended solid determinations are compared in Table 9.11 for Port S-3 and S-56. Due to the wide variation in the results, another method for determining suspended solids was considered. For both locations, the XRF analysis of the filters was tabulated minus the chloride concentrations; it can be assumed that the chloride is residue salt.

The weight of suspended solids was also determined by converting the laser data in particles per milliliter to milligrams per liter (see Table 9.12). Several assumptions were needed: the particles are perfect spheres, and the density of these particles is  $3\text{g/cm}^3$  (a mixture of silica and sulfides). It is interesting to note that for both Ports S-3 and S-56, the largest sized size fraction contained the largest mass fraction. As can be seen in Table 9.11, the total weight of suspended solids determined using the data obtained from the laser particle counter is in good agreement with the filter-weight-gain method for Port S-3, but high for Port S-56. The laser data may have been higher because: 1) bubbles were still forming in the brine during counting;

TABLE 9.11. Comparison of the Different Methods For Suspended Solid Determinations For Both One- and Two-Phase Brine

Method	One-Phase Brine (Port S-3) Suspended Solids (mg/l)	Two-Phase Brine (Port S-56) Suspended Solids (mg/l)
1. Filter-Weight-Gain		
a. Single Filter	0.26	0.77
b. Filter Chain	1.15	1.83
c. Filter Chain (hot)	8.48	--
d. Total XRF Data	0.02	0.36
2. Laser Particle Counter	0.77	32.9
3. Turbidity (FTU)	0.23	0.57

TABLE 9.12. Converting Laser Counts Into Mass Data

Size Range ( $\mu$ )	$\bar{r}^3$	Port S-3 Sample B-01		Sample B-11		Port S-56 Sample B-10		Sample B-12	
		Counts (Particles Per ml)	Mass (g) <sup>(a)</sup>	Counts (Particles Per ml)	Mass (g) <sup>(a)</sup>	Counts (Particles Per ml)	Mass (g) <sup>(a)</sup>	Counts (Particles Per ml)	Mass (g) <sup>(a)</sup>
1-5	15.6	534	$1.31 \times 10^{-8}$	356	$8.72 \times 10^{-9}$	351	$8.60 \times 10^{-9}$	369	$9.04 \times 10^{-9}$
5-10	422	72	$4.77 \times 10^{-8}$	359	$2.38 \times 10^{-7}$	272	$1.80 \times 10^{-7}$	367	$2.43 \times 10^{-7}$
10-15	1,953	21	$6.44 \times 10^{-8}$	236	$7.24 \times 10^{-7}$	231	$7.08 \times 10^{-7}$	242	$7.42 \times 10^{-7}$
15-20	5,359	7	$5.89 \times 10^{-8}$	130	$1.09 \times 10^{-6}$	140	$1.18 \times 10^{-6}$	143	$1.20 \times 10^{-6}$
20-25	11,391	4	$7.15 \times 10^{-8}$	88	$1.57 \times 10^{-6}$	94	$1.68 \times 10^{-6}$	93	$1.66 \times 10^{-6}$
25-30	20,797	2	$6.53 \times 10^{-8}$	61	$1.99 \times 10^{-6}$	59	$1.93 \times 10^{-6}$	56	$1.83 \times 10^{-6}$
30-35	34,328	1	$5.39 \times 10^{-8}$	44	$2.37 \times 10^{-6}$	37	$1.99 \times 10^{-6}$	39	$2.10 \times 10^{-6}$
35-40	52,734	0	--	31	$2.57 \times 10^{-6}$	27	$2.24 \times 10^{-6}$	25	$2.07 \times 10^{-6}$
40-45	76,766	0	--	25	$3.01 \times 10^{-6}$	22	$2.65 \times 10^{-6}$	17	$2.05 \times 10^{-6}$
45-50	107,172	1	$1.68 \times 10^{-7}$	17	$2.86 \times 10^{-6}$	14	$2.36 \times 10^{-6}$	12	$2.02 \times 10^{-6}$
50-55	144,703	1	$2.27 \times 10^{-7}$	16	$3.63 \times 10^{-6}$	13	$2.95 \times 10^{-6}$	11	$2.50 \times 10^{-6}$
55-60	190,109	0	--	13	$3.88 \times 10^{-6}$	7	$2.09 \times 10^{-6}$	7	$2.09 \times 10^{-6}$
60-65	244,141	0	--	11	$4.22 \times 10^{-6}$	6	$2.30 \times 10^{-6}$	7	$2.68 \times 10^{-6}$
65-70	307,547	0	--	9	$4.35 \times 10^{-6}$	5	$2.41 \times 10^{-6}$	5	$2.41 \times 10^{-6}$
70-75	381,078	0	--	6	$3.59 \times 10^{-6}$	4	$2.39 \times 10^{-6}$	5	$2.99 \times 10^{-6}$
75-80	465,484	0	--	6	$4.39 \times 10^{-6}$	3	$2.19 \times 10^{-6}$	3	$2.19 \times 10^{-6}$
			$76.98 \times 10^{-8}$ g/ml		$40.56 \times 10^{-6}$ g/ml		$29.26 \times 10^{-6}$ g/ml		$28.78 \times 10^{-6}$ g/ml

Average Port S-56 (32.9 mg/ml)

Port S-3 ( $76.98 \times 10^{-8}$  g/ml)(1,000 ml/l) =  $7.70 \times 10^{-4}$  g/l or 0.77 mg/lPort S-56 ( $32.9 \times 10^{-6}$  g/ml)(1,000 ml/l) =  $3.29 \times 10^{-2}$  g/l or 32.9 mg/l(a) Conversion factor:  $M = (1.57 \times 10^{-12}) \bar{r}^3$  (particles/cc), see Appendix A.

2) the particle density was actually lower than the  $3 \text{ g/cm}^3$  used for the calculations; and 3) the particle shapes were more varied from the ideal sphere volume used in the calculations. It is clear that while the laser particle counter gives much useful data, more work is needed to calibrate the mass analyses.

A probable reason that the XRF results are lower than the filter and laser results is that not all of the elements were analyzed, for example, oxygen, and some of the particulars, such as silica, exist as an oxide.

The comparison of each method for determining suspended solids in terms of time, cost advantages, and potential problems is summarized in Table 9.13.

## 9.6 CONCLUSIONS

All of the suspended solids measurement methods provided useful information. Further work will be required to define the most accurate method and the detailed procedures for standardization.

**TABLE 9.13. Summary and Comparison of Different Methods for Suspended Solid Determinations**

Method	Equipment Cost (\$)	Data Obtained	Advantages of Method	Potential Disadvantages
1. Filtration				
a. Single Filter	75	mg/l Solids	<ol style="list-style-type: none"> <li>1. Least inexpensive</li> <li>2. Suspended solids are collected to be analyzed by other methods (XRD, XRF, optical)</li> </ol>	<ol style="list-style-type: none"> <li>1. Need to correct for salt</li> </ol>
b. Filter Chain, Cold Sampling	3,000	mg/l solids, plus particle size distribution	<ol style="list-style-type: none"> <li>1. Suspended solids for each size range are collected for study (XRD, XRF, optional)</li> </ol>	<ol style="list-style-type: none"> <li>1. Need to correct for salt</li> </ol>
c. Filter Chain, Hot Sampling	3,000	mg/l solids, plus particle size distribution	<ol style="list-style-type: none"> <li>1. Suspended solids are collected of each size range for further study</li> <li>2. Sampling at in-line conditions</li> </ol>	<ol style="list-style-type: none"> <li>1. Need to correct salt</li> <li>2. Must be careful flashing does not occur</li> </ol>
d. Summation of XRF Data on Filter	Need an x-ray Spectrometer, which is very expensive	$\mu\text{g}/\text{cm}^2$ of each element collected, and $\mu\text{g}/\text{l}$ if volume of brine that passed through filter is known	<ol style="list-style-type: none"> <li>1. Reveals chemistry of suspended solids</li> </ol>	<ol style="list-style-type: none"> <li>1. Does not analyze all elements (ex. oxygen), so weight will be low</li> </ol>
2. Laser				
a. Laser Particle Counter	8,200	Total particles/ml	<ol style="list-style-type: none"> <li>1. Can determine variations in suspended solids over time</li> <li>2. Least time and data manipulation</li> </ol>	<ol style="list-style-type: none"> <li>1. To convert to mg/l, need to estimate the density and assume the shape of the suspended solids</li> </ol>
b. Counter Plus Profile Attachment	13,700	Total particles/ml plus particle size distribution, and total mass (mg/l) plus mass distribution	<ol style="list-style-type: none"> <li>1. Can determine variations in suspended solids over time</li> <li>2. Least time and data manipulation</li> </ol>	<ol style="list-style-type: none"> <li>1. To convert of mg/l, need to estimate the density and assume the shape of the suspended solids</li> </ol>
3. Turbidity	600	Turbidity of fluid in various turbidity units	<ol style="list-style-type: none"> <li>1. Inexpensive</li> </ol>	<ol style="list-style-type: none"> <li>1. The units are not directly relatable to mg/l</li> </ol>

## 9.0 REFERENCES

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2. "Filter Clearing Procedure For Particle Counting Using Transmitted Light." Millipore Technical Brief TS018, Millipore Corporation.
3. J. Grens. "Preliminary Tests Using a Laser Particle-Size Analyzer on Geothermal Brine." Lawrence Livermore Laboratory, UCID-17637, 1977.

## 10.0 MEASUREMENT OF NONCONDENSIBLE GASES (TEST 8)

D. W. Shannon

### 10.1 WHAT ARE NONCONDENSIBLE GASES?

The collection of a geothermal liquid sample from pressurized systems is usually accompanied by various amounts of gas. The gas is often described as "noncondensable" to distinguish it from "condensable gases" which is usually steam.

The measurement of noncondensable gases is of interest to turbine designers since, when the steam-gas mixture is passed through a turbine, the steam can be condensed to liquid, whereas the gases cannot. The buildup of gases in the condenser systems raises the turbine back pressure, seriously degrading turbine performance. Such gases must be removed by vacuum pumping systems or by steam eductors, which can be a significant parasitic power load on the power plant. A geothermal resource with a very high noncondensable gas content could be less attractive to develop than a resource with a low gas content. In any event, the designer must know or be able to measure gas content of a resource.

### 10.2 THE DIFFICULTIES OF NONCONDENSIBLE GAS SAMPLING

CO<sub>2</sub> usually composes the bulk of noncondensable gases from geothermal resources. The gases can also contain significant levels of CH<sub>4</sub>, N<sub>2</sub>, H<sub>2</sub>, Ar, H<sub>2</sub>S, C<sub>2</sub>H<sub>6</sub>, and NH<sub>3</sub>. Occasionally, O<sub>2</sub> is present from contamination by sampling equipment, from leaking seals, or maintenance operations on piping systems, but O<sub>2</sub> does not usually exist in the reservoir in the strongly reducing environment of geothermal waters.

The problem in sampling is that these gases are soluble in the geothermal water, and solubilities are complicated functions of temperature, pressure, salinity, pH, and brine composition. When the fluids are depressurized during sampling, the gas bubbles break out of solution like they do when a bottle of champagne is opened. The amount of gas released depends on sampling conditions such as temperature, pressure, residence time, agitation, and pipe geometry,

as well as brine chemistry effects. In some cases, gases are dissolved in the liquid instead of released because higher solubilities are present at ambient temperature.

Not only is the amount of gas released per kg of brine quite variable, but the percent composition of various gas components can vary due to variations in CO<sub>2</sub> release, which dilutes other components. A common example is that CO<sub>2</sub> release varies with degree of flashing of steam. This can be seen in Table 10.1, where the CO<sub>2</sub> content of the gas from East Mesa 6-2 was 97.75% when the sample was taken under flashing conditions, compared with 90.02% when the sample was obtained by depressurizing a single-phase sample. The greater CO<sub>2</sub> release dilutes the other species, which causes their percentage analysis to drop.

TABLE 10.1. Change in Gas Composition Due to Sampling Method and Location (mole % by Mass Spectrometer)

	<u>CO<sub>2</sub></u>	<u>Ar</u>	<u>O<sub>2</sub><sup>(a)</sup></u>	<u>N<sub>2</sub></u>	<u>H<sub>2</sub></u>	<u>CH<sub>4</sub></u>	<u>C<sub>2</sub>H<sub>6</sub></u>
<u>Nonflashing Conditions</u>							
Gas sample from top of Port 5, Brine 167C, 125 psi, 8/22/80	90.01	0.06	0.01	2.56	0.08	7.09	0.19
<u>6% Steam Flash Conditions</u>							
Gas sample from top of Port 56 using Webre separator, Brine 139C, 49 psi, 8/22/79	94.64	0.04	0.04	1.18	0.05	3.92	0.13
Gas sample from Port 57 into evacuated cylinder, Brine 136C, 40 psi, 8/21/79	81.6	0.08	0.14	4.76	0.03	13.26	0.17

(a) Traces of O<sub>2</sub> are due to contamination of samples with air during sampling, airline transport, or analytical procedure.

Problems arose with the sample produced by expanding the geothermal fluids into an evacuated stainless steel cylinder (see Table 10.1). Since there is cold brine in the cylinder, the  $\text{CO}_2$  redissolves, which reduces the  $\text{CO}_2$  content of the gas and increases the percentage of other less soluble gases.

The problem of gas sampling is complicated further if the system is two-phase steam-gas and liquid because it is highly likely that the gas bubbles are not uniformly distributed. And even if the gas bubbles are evenly distributed, gas-to-liquid ratios are difficult to pull into the sample probe unless isokinetic sampling probes are reset (see Section 13). Gas bubbles may exist in the two-phase system, but dissolve back into the liquid upon cooling in the sampling system. Hot pressurized separators may be needed in this case.

### 10.3 SAMPLING TWO-PHASE $\text{CO}_2$ -WATER MIXTURES

#### 10.3.1 Equipment

The first tests were to determine gas-to-liquid ratios just downstream of the first loop orifice, where the pressure had dropped from 125 psi to 100 psi, producing a " $\text{CO}_2$  flash", but little (<1%) steam flash. Two sampling probes of 1/4" stainless steel tubing and 1/2" stainless steel tubing were used in adjacent Ports 20 and 22. Both probes were vertical to permit vertical traverses from the gas cap on the top of the pipe to the almost pure liquid on the bottom.

The probe was connected to double cooling coils to remove heat, and then the coil outlet was passed to a glass separator (see Figure 10.1). The gas from the glass separator was run through a wet test meter and the liquid was discharged into a 2-liter graduated cylinder. The amount of gas collected was integrated over a 5 min period; and liquid was measured at the same time to give a gas-to-liquid ratio.

For purposes of comparison, we also measured gas/liquid ratios using a glass bulb method (Figure 10.2). A glass bulb (total volume 0.725l) was filled with brine by attaching it with Tygon<sup>®</sup> tubing to the coil outlet,

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<sup>®</sup>Tygon tubing.

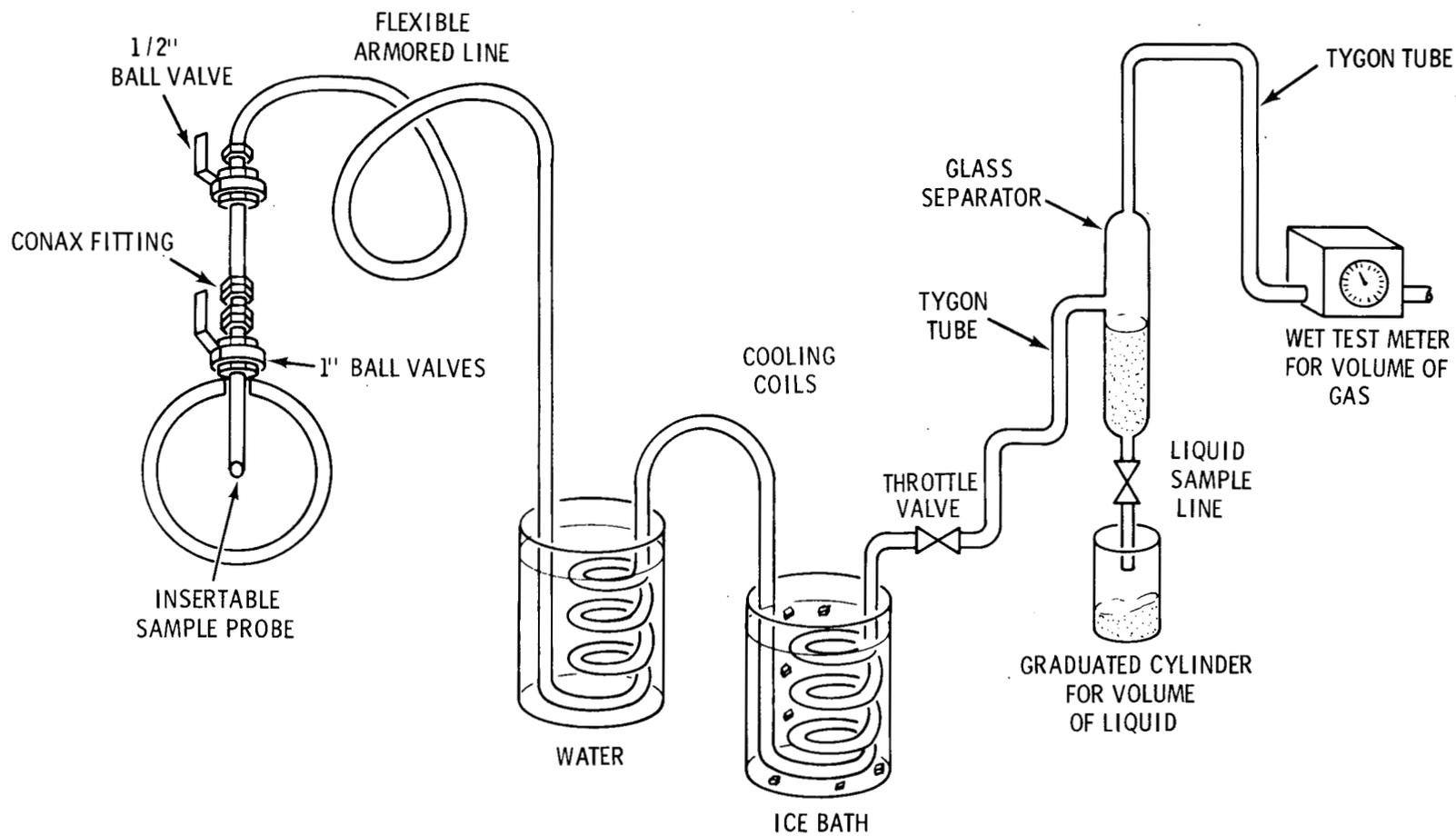


FIGURE 10.1. Wet Test Meter Method to Determine Gas/Liquid Ratios

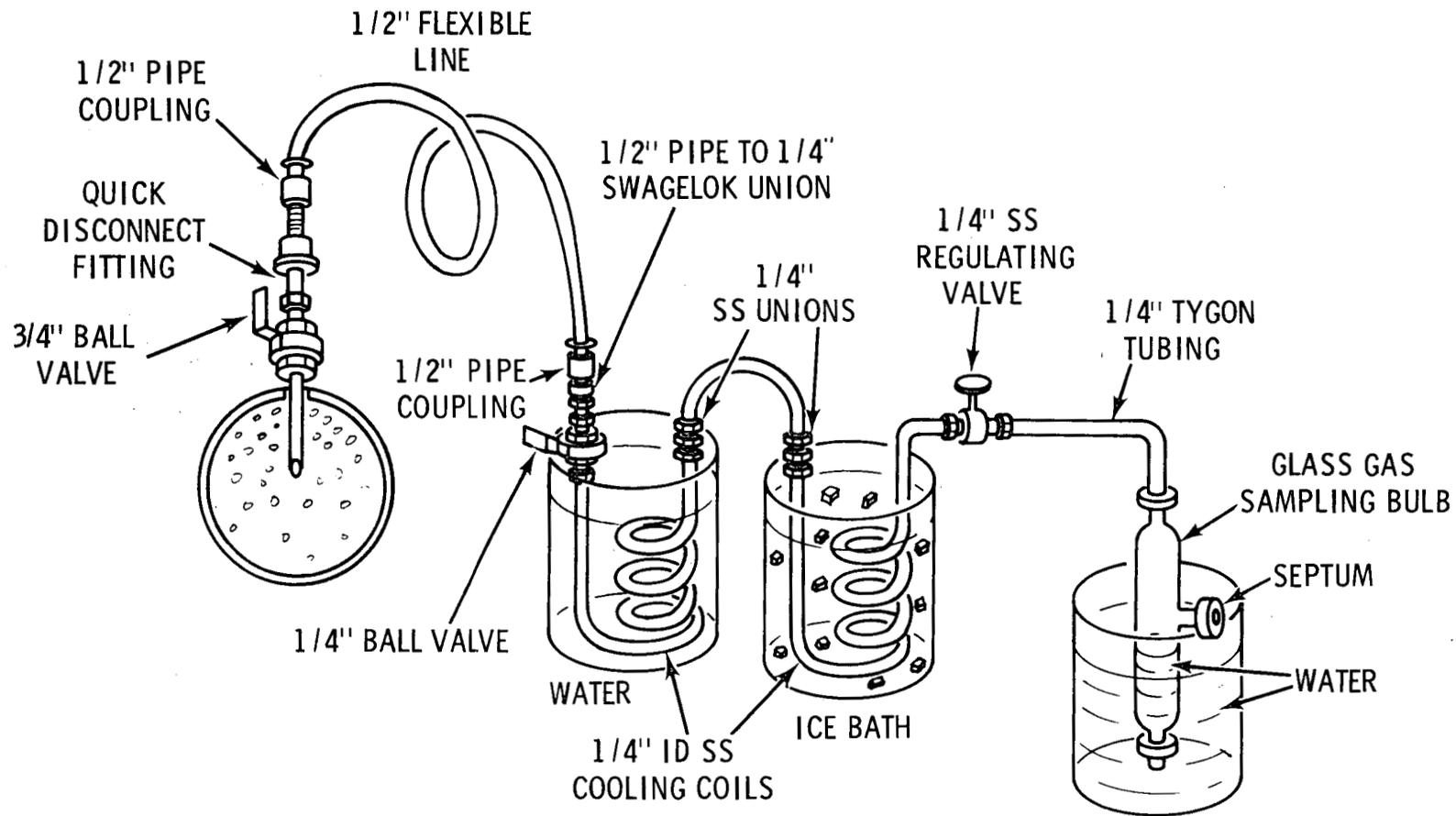


FIGURE 10.2. Method of Collecting Gas Samples

initially holding the bulb with the outlet up to permit all gas to escape. At time zero, the bulb was inverted to permit liquid to flow into a 1-liter graduated cylinder. When exactly 1 liter had been collected, flow was stopped, the bulb removed from the coil outlet and the remaining brine in the bulb drained into a cylinder to measure its volume (V). The gas associated with 1 liter of brine was calculated:

$$0.725\ell - V = \text{Vol. Gas}; \quad \frac{\text{Vol. Gas}\ell}{1\ell \text{ Liquid}} = \text{ratio}$$

Gas samples for composition analysis are taken by a similar bulb method except the entire bulb is filled with gas as shown in Figure 10.2.

### 10.3.2 Results

The first measurements were designed to see if the measured gas:liquid ratio was sensitive to flow. By adjusting the regulating valve, various flow rates were obtained and gas:liquid ratios calculated. These results in Table 10.2 show that the gas:liquid ratio was sensitive to sample flow rate, probe diameter, and probably to temperature. (It was not possible to increase flow without increasing temperature, too.) The gas:liquid ratios varied over a factor of 20, which is not precise enough to be useful.

TABLE 10.2. Gas Liquid Ratios at Various Flow Rates Using Glass Separator

1/4" Dia. Sample Probe 1" From Top Of Pipe			1/2" Dia. Sample Probe 1" From Top of Pipe			
Liquid Rate ℓ/m	Gas Rate ℓ/m	Gas/Liquid	T °C	Liquid Rate ℓ/m	Gas Rate ℓ/m	Gas/Liquid
0.014	0.016	1.14	17	0.12	0.11	0.97
0.086	0.077	0.90	29	0.23	0.056	0.24
0.13	0.17	1.27	39	0.80	0.75	0.92
0.28	1.17	4.58	39	0.79	0.79	1.01
0.32	0.84	2.6	37	0.76	0.61	0.79
0.44	1.48	3.37	53	1.10	1.36	1.23
0.45	1.43	3.15				
0.79	4.44	5.63				

The 1/4"-diameter probe gave higher gas:liquid ratios than the 1/2"-diameter probe at equal flow rates. At equal flow rates, there is a greater pressure drop between the bulk fluid and the probe inside for the 1/4" probe, favoring gas enrichment. Clearly, an isokinetic probe is needed (see Section 13). As expected, the gas:liquid ratio tended to increase with increasing flow since the lower internal probe pressure could favor collection of gas. In this case, the temperature of the liquid also rose, leading to greater gas breakout.

The gas:liquid ratio was also determined with the bulb method illustrated in Figure 10.2. The results, given in Table 10.3, show excellent precision, but the factor of 2 difference between 1/4" probes and 1/2" probes clearly illustrates that the probes are not correctly determining the gas:liquid ratio in the pipe. A comparison of the data on the glass bulb method in Table 10.3 with the separator method in Table 10.2 (at the same flow of about 0.4ℓ/min) shows the bulb method produces lower gas:liquid ratios by a factor of 2 or more. This probably is due to the fact that the gas is redissolving in the liquid. The much longer gas/liquid contact times present in the bulb method permit more gas to dissolve.

The problem of gas solubility in the liquid is dramatically illustrated by the data in Table 10.4. These samples were taken in the flashing zone downstream of the second orifice. In this zone, the hot liquid has a pH of about 8.5, and CO<sub>2</sub> easily dissolves during sampling. Depending on sampling temperature, CO<sub>2</sub> dissolves, in differing amounts, leading to extreme variations in gas composition, all from the same port!

TABLE 10.3. Gas Liquid Ratios Determined by Bulb Method  
(Temperature of Liquid, 26°C±1; Liquid Flow  
Rate, 4 ℓ/min for both 1/4" and 1/2" Probes)

Triplicate Determinations, 1/4" Probe			Triplicate Determinations, 1/2" Probe		
Liquid Collected (ℓ)	Gas Collected (ℓ)	G/L Ratio	Liquid Collected (ℓ)	Gas Collected (ℓ)	G/L Ratio
1.02	0.682	0.66	1.04	0.345	0.33
1.02	0.680	0.67	1.04	0.420	0.40
1.13	0.671	0.65	1.02	0.335	0.33

TABLE 10.4. The Effect of Sampling Temperature on the Composition of Gas Samples (Samples Collected From Top at Port 76 on 8/27/79, Brine 139C, 45 psi with 6% Steam Flash)

	Mole %		
	<u>Collected at 15°C</u>	<u>Collected at 25°C</u>	<u>Collected at 52°C</u>
CO <sub>2</sub>	32.1	75.84	86.83
Ar	0.45	0.16	0.08
O <sub>2</sub>	3.32 <sup>(a)</sup>	0.86	0.05
N <sub>2</sub>	23.93	7.68	3.55
H <sub>2</sub>	2.00	0.21	0.13
CH <sub>4</sub>	37.38	14.93	9.06
C <sub>2</sub> H <sub>6</sub>	0.29	0.32	0.26

(a) Note: O<sub>2</sub> is due to sample contamination. Because of low CO<sub>2</sub> breakout, total gas rate was low, making it difficult to pressurize collection bulb.

### 10.3.3 Conclusions

Gas/liquid sampling of a two-phase pipe is subject to so many unknown variables that it is almost impossible to be sure you have the "right" answer.

We recommend that the full flow of the pipe be directed into a full flow separator and that pressure or temperature drops be kept at a minimum to prevent further flashing). We also recommend that single-phase sampling methods presented in this report be used on the separate gas and liquid phases. If the flow rates of the gas and liquid streams are known, a gas:liquid ratio can be calculated.

The measurement of gas:liquid ratios is discussed further in the chapter on sampling with separators (Section 11).

## 11.0 SAMPLING TWO-PHASE SYSTEMS WITH SEPARATORS (TEST 10)

D. W. Shannon

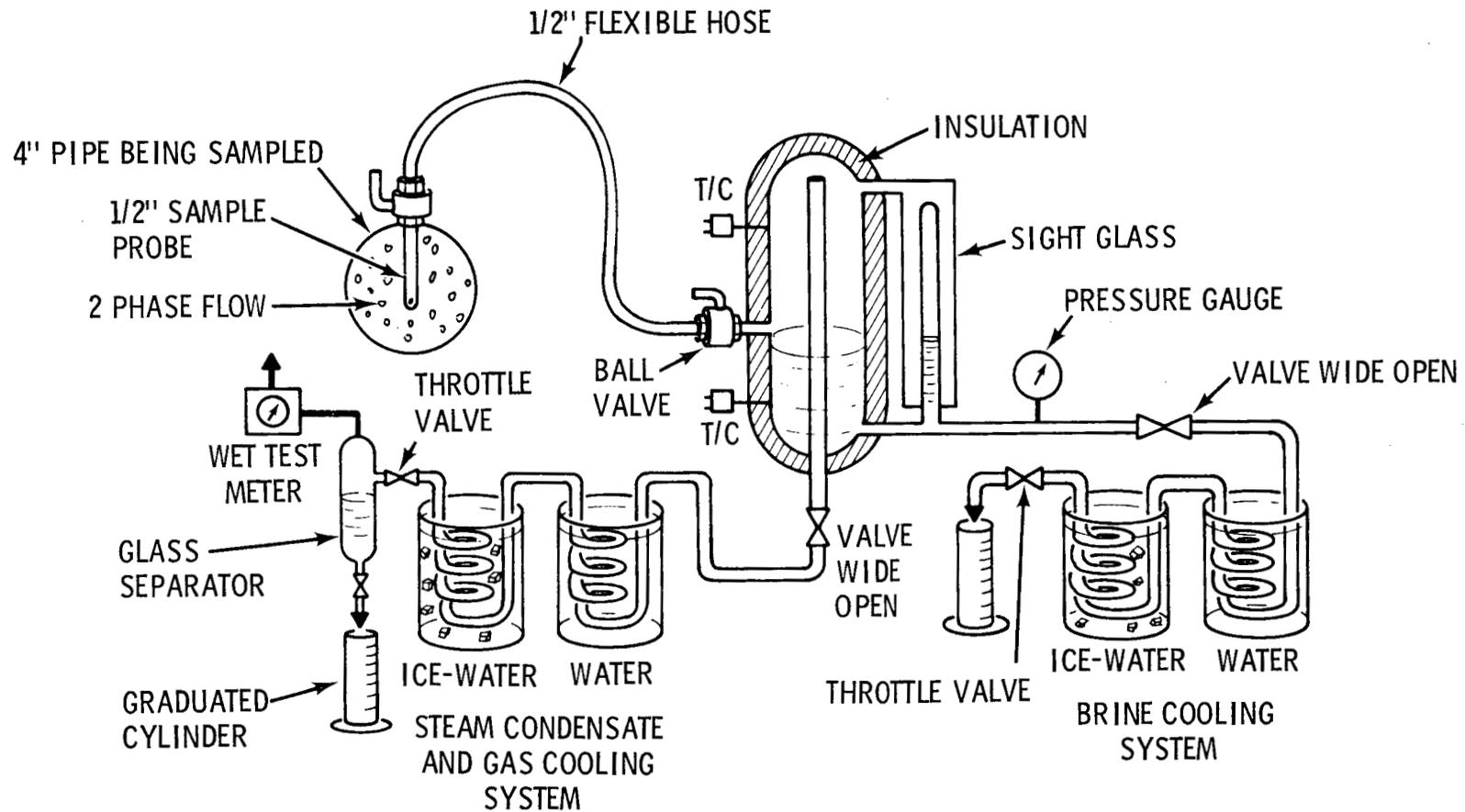
### 11.1 OBJECTIVE

The objectives of this test were to use a traversing probe to remove representative samples of steam/gas plus liquid at loop pressure and to determine if the resulting data could be "reassembled" to estimate the actual composition flowing in the pipe. Further flashing at the sample was prevented by operating an insulated small separator at loop temperature and pressure. This permitted measurements of the steam, brine and gas composition existing in the two-phase region at the sample tube inlet (subject to nonisokinetic probe errors).

### 11.2 EQUIPMENT

A small Webre separator was used as shown in Figure 11.1. A 1/2" dia. sample probe was inserted through a packing gland into the loop and connected to the separator with a 1/2" flexible braided hose and 1/2" ball valve. The separators operated at full system pressure and were insulated to minimize cooling. Skin thermocouples were attached to measure temperatures and sight glass was provided for level control.

Two sets of double sampling coils were used; one set on the steam line and one set on the brine line. The output of the condensed steam line which contained gas was further routed to a glass separator to separate the gases from the condensate using methods described in Section 10. At the start of sampling at a given point, the inlet ball valve to the separator was opened wide to pressurize the separator with both the steam condensate and brine throttle valves closed. The steam throttle valve was then opened to bleed off gases, and to start condensing steam at a rate within the capacity of the cooling system. The liquid level in the Webre separator would begin to rise as steam was released and more liquid/steam entered. The brine outlet throttle valve was then gradually opened to hold the liquid level constant, adjusting every few minutes as required to maintain constant level. After all temperatures and flows stabilized, a set of readings and samples were taken.



**FIGURE 11.1.** Small Webre Separator Used to Sample Steam, Gas, and Liquid At System Temperature and Pressure

A single vertical traverse was run at 9 locations of the 4" loop pipe using Port 56 just downstream of the second flashing orifice in the two phase steam-brine gas region. This traverse took about 5 hours to conduct. During this time, calcium carbonate scale was forming in the loop causing pressures in the loop to slowly rise.

Because of the very large number of samples, no attempt was made to analyze for all species. Instead, we selected pH, total  $\text{CO}_2$  to measure  $\text{CO}_2$  gas distribution,  $\text{Na}^+$  and  $\text{Cl}^-$  to measure salinity changes due to steam/brine ratios, and  $\text{Ca}^{+2}$  for a chemical species that was actively forming scale. Several gas composition bulb samples were also taken.

### 11.3 RESULTS

The operating data and brine, condensate, and gas flow rates are given in Table 11.1. The flow rates from the separator showed the top of the pipe contained only steam with a large amount of gas, whereas the bottom of the pipe contained only brine containing little gas.

The chemical data for the brine and condensate are given in Table 11.2. These data clearly show the release of  $\text{CO}_2$  from the brine during flashing, enriching the steam phase in  $\text{CO}_2$ . The chloride data for the brine show the effect of steam loss increasing salinity of the brine near the bottom of the pipe. It should be kept in mind that there was only a 6% by weight total steam flash, so large concentration changes would not be expected.

In Table 11.3, we have recombined the  $\text{CO}_2$  data from the three sources, brine, condensate and gas, in order to compute a total  $\text{CO}_2$  per liter of brine plus condensate. These data clearly show the gradient of  $\text{CO}_2$  from the bottom to the top of the pipe cross-section.

If each zone of the pipe were moving at the same velocity, and the gradient from top to bottom simple represented a redistribution of the  $\text{CO}_2$ , it should be possible to calculate a weighted average  $\text{CO}_2$  based on pipe cross-sectional area to arrive at the average  $\text{CO}_2$  content of the total flow. This has been calculated in Table 11.4.

TABLE 11.1. Operating Conditions and Separator Output During Probe Traverse

Probe Distance From Pipe Bottom (in)	Loop Conditions At TP13		Separator Conditions		Flow Rates From Separator (ℓ/min)			
	T (°C)	P (psi)	T (°C)	P (psi)	Brine Rate	Condensate Rate	% Steam (a)	Gas Rate (b)
4 (top)	142	49	145	54	0	0	100	3.46
3 1/2	141	50	144	52	0.43	0.16	27	3.25
3	140	44	143	50	0.75	0.13	15	2.28
2 1/2	138	46	143	50	0.88	0.085	8.8	1.19
2	138	43	143	49	0.85	0.036	4.0	0.46
1 1/2	138	42	142	49	1.11	0.030	2.6	0.32
1	136	43	142	48	0.92	0.014	1.5	0.23
1/2	135	43	140	47	0.50	0.005	0.1	0.08
0 (bottom)	135	40	139	47	2.0	0.005	0.02	0.21

(a) Percent of water as steam, calculated by:  $\frac{\text{condensate rate}}{\text{brine rate} + \text{condensate rate}} \times 100$ .

(b) Mass spec. analysis, -94% CO<sub>2</sub>.

TABLE 11.2. Chemical Composition of Brine and Condensate From Webre Separator

Probe Distance From Pipe Bottom (in)	Brine Composition mg/ℓ					Condensate Composition mg/ℓ		
	pH	Total CO <sub>2</sub>	Cl <sup>-</sup>	Na <sup>+</sup>	Ca <sup>+2</sup>	pH	Total CO <sub>2</sub>	Na <sup>+</sup>
4 (top)	--	--	--	--	--	5.5	--	<2
3 1/2	8.5	370	1960	1450	2.3	5.2	2234	<2
3	8.4	357	1961	1483	2.0	5.2	2169	<2
2 1/2	8.5	361	1987	1500	2.8	5.5	--	--
2	8.6	368	1581	1350	2.0	5.5	--	7
1 1/2	8.7	405	1983	1484	2.1	5.5	--	8
1	8.7	405	1983	1484	2.1	5.5	--	337
1/2	8.5	374	1992	1400	2.3	5.5	--	308
0 (bottom)	8.4	436	2002	1450	3.0	5.5	--	13

TABLE 11.3. Mathematical Summation of CO<sub>2</sub> in Brine, Steam and Gas  
(CO<sub>2</sub> in Brine + CO<sub>2</sub> in Steam Condensate + CO<sub>2</sub> in Gas  
Phase = Total CO<sub>2</sub> (mg/l))

Location	Total CO <sub>2</sub> in Brine (mg/l)	Total CO <sub>2</sub> in Steam Cond. (mg/l)	l Gas <sup>(a)</sup>	mg CO <sub>2</sub> <sup>(b)</sup>	Total CO <sub>2</sub> (mg/l) <sup>(c)</sup>
4 (top)	0 = 0	2200 x 1.0	20.3	34,695	36,895
3 1/2	370 x .73 = 270	2200 x .27 = 594	5.51	9,417	10,281
3	357 x .85 = 303	2200 x .15 = 330	2.59	4,427	5,060
2 1/2	361 x .97 = 329	2200 x .09 = 198	1.22	2,085	2,612
2	368 x .96 = 353	2200 x .04 = 88	0.52	889	1,330
1 1/2	339 x .97 = 328	2200 x .03 = 66	0.28	479	873
1	400 x .98 = 401	2200 x .02 = 44	0.24	410	855
1/2	374 x .99 = 370	2200 x .01 = 22	0.16	273	665
0 (bottom)	436 x 1.00 = 436	0 = 0	0.11	188	624

(a) Calculated by  $\frac{\text{l/min gas}}{\text{l/m brine} + \text{l/m condensate}}$

(b) Calculated by  $\text{CO}_2 \text{ mg} = \frac{\text{l gas}}{\text{l liquid}} \times .94$   
 $\frac{24.2 \text{ (at 298K)}}{44,000}$

(c) Calculated by  $\text{CO}_2(\text{l}) + \text{CO}_2(\text{cond}) + \text{CO}_2(\text{gas})$ .

TABLE 11.4. Calculation of Weighted Average CO<sub>2</sub> Content

<u>Zone</u>	<u>Area of Each Each Zone (in<sup>2</sup>)</u>	<u>Fraction of Area in Each Zone</u>	<u>CO<sub>2</sub> Content mg/l</u>	<u>Weighted CO<sub>2</sub> Content of Zone mg/l</u>
4	0.32	0.025	36,895	830
3 1/2	1.31	0.1046	10,281	1,075
3	1.73	0.138	5,060	698
2 1/2	1.93	0.154	2,612	402
2	1.98	0.158	1,330	210
1 1/2	1.93	0.154	873	134
1	1.73	0.138	855	118
1/2	1.31	0.105	665	70
0	.32	0.025	624	16
			Total	3,553

The area weighted average CO<sub>2</sub> content determined this way calculates to 3,553 mg/l. The original brine at the test loop inlet before flashing contamination was 1,087 mg/l or one third as much. This means that either there was a static gas cap in the pipe (which is very possible) or that the sample probe used was enriching the mixture in gas (which is also possible). It would take much more data to prove why the CO<sub>2</sub> content is so high.

Another set of data were obtained by flashing the inlet brine from Port 5 into a single stage separator at the same temperature and pressure as was found in the two phase region of the test loop in the above example. These data are given in Table 11.5 where the single flash data (Part B) are compared with single separator data from a probe in the center of the stream using Port 55. These data are compared with the single phase unflashed brine which was at the test inlet. Comparisons of the data show that after the corrections for flash, the single stage flash analytical data compare favorably with the control data for most species except calcium and strontium which have been lost due to carbonate scaling as flashing occurred. These results indicate it is possible to calculate a composite composition by sampling both the

TABLE 11.5. Mathematical Recombination of Data From Two Phase Sampling Using Separators (Concentration in mg/l)

	A	B			C		
	Inlet Brine Composition Single Phase At Port 3, T = 167C, P = 132 psi	Stage Flash Using Same Brine as A, T = 137C, P = 132 psi			Two Phase Flow Zone Center of Port 55, Separator Data, T = 137C, P = 40 psi		
	Single Phase	Condensate	Brine	Corrected Brine(a)	Condensate	Brine	Corrected Brine(a)
pH	5.8	5.4	8.3	--	5.8	8.2	--
Field NH <sub>3</sub>	13	80	15	18	100	15	17
Field H <sub>2</sub> S	0.4	3	0.3	0.32	1	0.3	0.33
TDS	4175	--	--	--	--	4410	4308
Total CO <sub>2</sub>	1155	1585	367	1192 <sup>(b)</sup>	2755	371	1492 <sup>(b)</sup>
Cl <sup>-</sup>	1901	--	2003	1902	--	2014	1968
SiO <sub>2</sub>	212	--	--	--	--	187	182
As	0.7	<0.01	0.7	0.67	0.2	0.6	0.58
B	7.5	0.54	7.9	7.5	2.0	8.0	7.8
Ba	0.3	0.01	0.2	0.2	0.1	0.2	0.2
Ca	8.5	3.2	2.4	2.3	3.8	2.5	2.8
Fe	0.2	0.8	0.1	0.1	0.2	<0.1	<0.1
Li	4.8	0.3	5.0	4.8	1.2	5.2	5.1
Mg	0.2	0.09	0.4	0.38	0.1	0.2	0.2
Na	1512	95	1545	1468	378	1610	1572
Si	101	1.7	105	100	26	108	105
Sr	2.1	.02	0.8	0.76	0.7	0.9	0.87
Separator Mass Flow Rates		0.005	0.98	0.031	1.33		
% Flash		5.2%			2.3%		

(a) Brine data corrected for observed steam flash although overall flash for Data C was 6%, not 2.3% observed.

(b) CO<sub>2</sub> data corrected by adding CO<sub>2</sub> in brine, condensate and gas.

single-phase steam/gas and brine phases from a full size separator. This method produces much more reliable results than attempting to withdraw representative samples from the two-phase region.

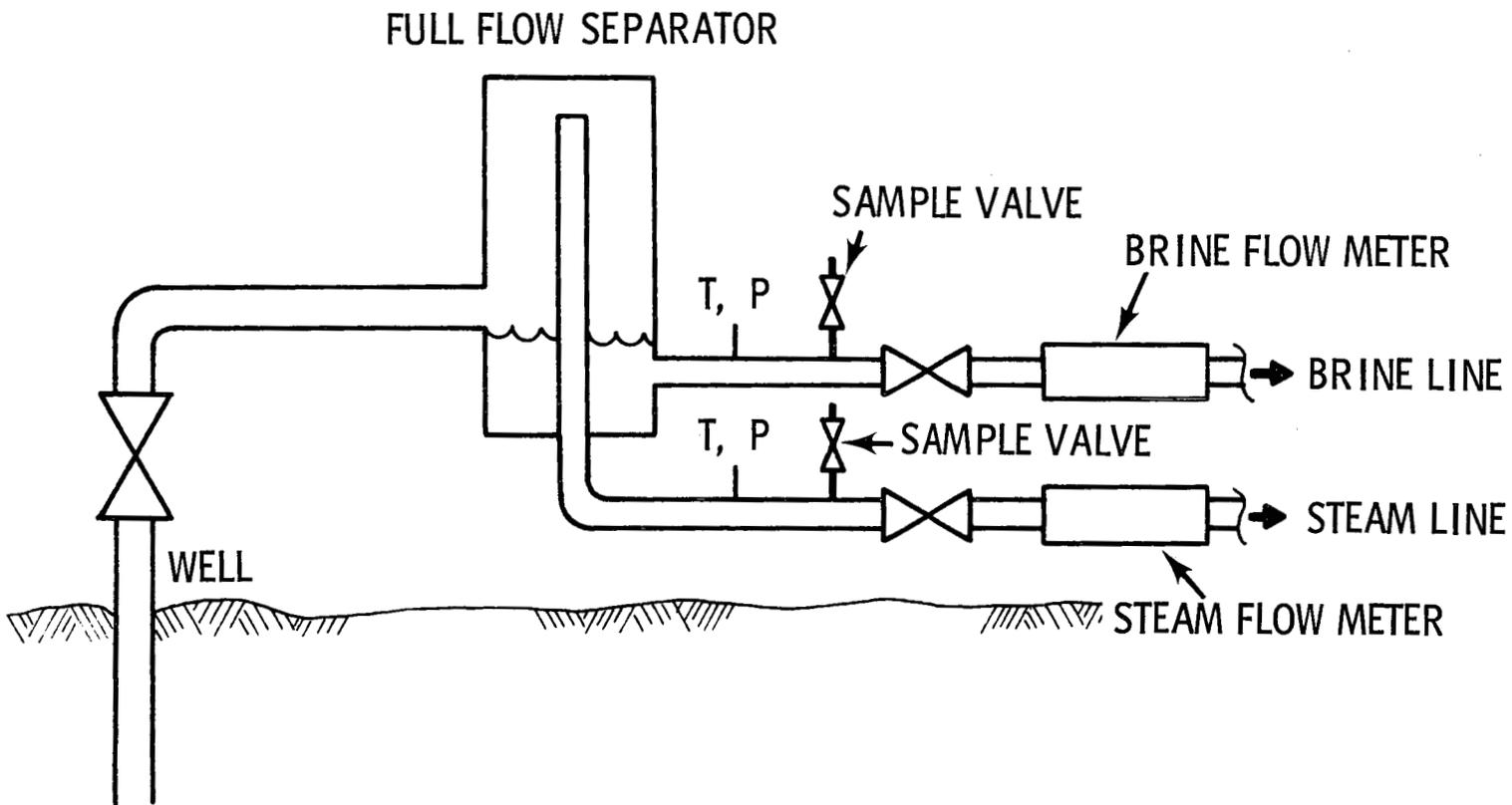
#### 11.4 CONCLUSIONS

It was not possible in these tests to sample the two-phase pipe and calculate the correct average composition. The small separator illustrated in Figure 11.1 provided the most consistent data, but the CO<sub>2</sub> values are too high. The separation of the gas and steam from the brine at system temperature and pressure without further flashing is the key point. This avoids drastic changes in gas/liquid ratios which can occur if the phases are cooled and depressurized in contact with each other. The method still suffers from the problem of nonuniform pick up of the "proper" ratio of steam/gas to liquid. This requires use of an isokinetic sampling probe discussed in the next section.

It may be possible to develop a two-phase sample system. This would utilize a mixer which would be put into the line to homogenize the flow which would be sampled with an isokinetic probe. The output of the isokinetic probe would be separated as in Figure 11.1. However, such a system would be quite expensive to develop and fabricate.

Until adequate two phase sampling systems are proven, we do not recommend taking samples from two-phase systems. The results are, at best, ambiguous, and probably will mislead users of the data. Our recommended method for handling two-phase systems is illustrated in Figure 11.2. The full flow of the well is passed into a full flow separator where the steam/gas is separated from the liquid. Both single-phase systems are sampled separately after the well has flowed for several days.

The total mass flow of the steam and liquid phases of the separator must be measured accurately, but this is often done as part of an evaluation of a new geothermal well production test. The resulting chemical analysis data can be recombined by adding together the total mass flow rates and compositions. The major deficiency will be that scaling species such as calcium will probably be lost during flashing resulting in low values.



## NOTE:

- (1) 1/2" DIAMETER PROBES FACING INTO THE FLOW ARE INSERTED INTO SAMPLE VALVES TO REACH THE CENTER OF THE PIPE.
- (2) DOUBLE COOLING COILS AND GAS MEASURING EQUIPMENT ARE ATTACHED TO PROBES AS USED IN FIGURE 10.1

FIGURE 11.2. Recommended Arrangement For Sampling Two-Phase Geothermal Wells

If the well will flow in a low flow artesian mode, it will be useful to collect a set of samples from this single phase system (again after flowing the well for several days). These single phase data will be especially useful for pH and species easily lost by scaling during flashing, such as calcium.

## 12.0 ISOKINETIC SAMPLING PROBE (TEST 11)

G. A. Jensen and J. R. Divine

### 12.1 INTRODUCTION

An isokinetic probe (I-probe) was designed and used to obtain samples of fluids at Port 84 of the 4 inch Schedule 46 Sampling Test Loop described in Chapter 3. The samples were taken at 1/2 inch intervals along the vertical centerline. This chapter describes the design, sampling procedure and results of this test.

Isokinetic sampling is not new and has been used by a number of investigators to measure flows and enthalpy.<sup>(1-5)</sup> It is a useful tool for sampling fluids in two-phase flow because samples so obtained are "representative" of the fluid mixture at the location where the sample is obtained. From Bernoulli's equation and a momentum balance, it can be shown that when the static and free stream static pressure are equal, isokinetic conditions exist at the sample port and a "representative" two-phase sample is obtained.<sup>(6)</sup> Static pressure taps in the sample stream and in the free stream are used to measure the static and free stream pressures at the sample inlet port.

The stated objectives of this work were to:

- Design and build an isokinetic probe (I-probe) for sampling geothermal fluids,
- Determine the operating characteristics of the I-probe,
- Compare samples obtained with the I-probe with those obtained using a standard sampling probe (R probe), and
- To establish if the isokinetic probe can be used to sample two-phase flow in pipes to "reconstruct" a "representative" composition.

Accordingly, it was necessary to determine whether:

- The current sampling procedures were compatible with the I-probe.
- The samples obtained represent the flow conditions existing at the profile location in the pipe where the sample was obtained and how these data relate to the total composition in the pipe.

All of the objectives and evaluations were met within the limits of data obtained. Laboratory analysis of samples and calculation of gas-steam-liquid ratios are all representative of the fluid character at the vertical location where the sample was obtained. The details of this effort are found in the following sections.

#### 12.1.1 Probe and Sample System Design

An outline drawing and a picture of the probe were found in Figures 12.1 and 12.2. The following constraints were placed on the I-probe design by the design of the 4 inch Sampling Test Loop and expected flow conditions. These were:

- The I-probe had to be sized for insertion through a 1 inch NPT welded half coupling on the 4 inch Sample Test Loop.
- The sampler had to be capable of accepting 1/8 to 1/4 inch liquid droplets without plugging the opening.
- The I-probe had to be self-contained to withstand system pressures; simplification of the design required that pressure tap lines pass out of the system inside the sampler.
- Probe had to access the pipe 90° to the direction of geothermal fluid flow, while sampling facing into the direction of flow.

The first and second of the above constraints may have compromised the performance of the probe. Normally a tapered section 5 to 8 port diameters long is specified in order to maintain the streamlines and reduce or eliminate turbulent effects on sampling. Because of the size requirement for the port, the tapered section had to be limited to between 1 to 2 port diameters with a 90° sharp bend in the probe just downstream of the end of the tapered section.

The free stream pressure tap and static pressure tap were contained within the sampling tube. Because the tubing for these pressure taps was contained within the flow area of the sampler, the sample port was sized to 11/32 inch in diameter to assure that the velocity in the entry section and the sample tube would remain constant. Other considerations such as probe length and general configuration were arbitrary.

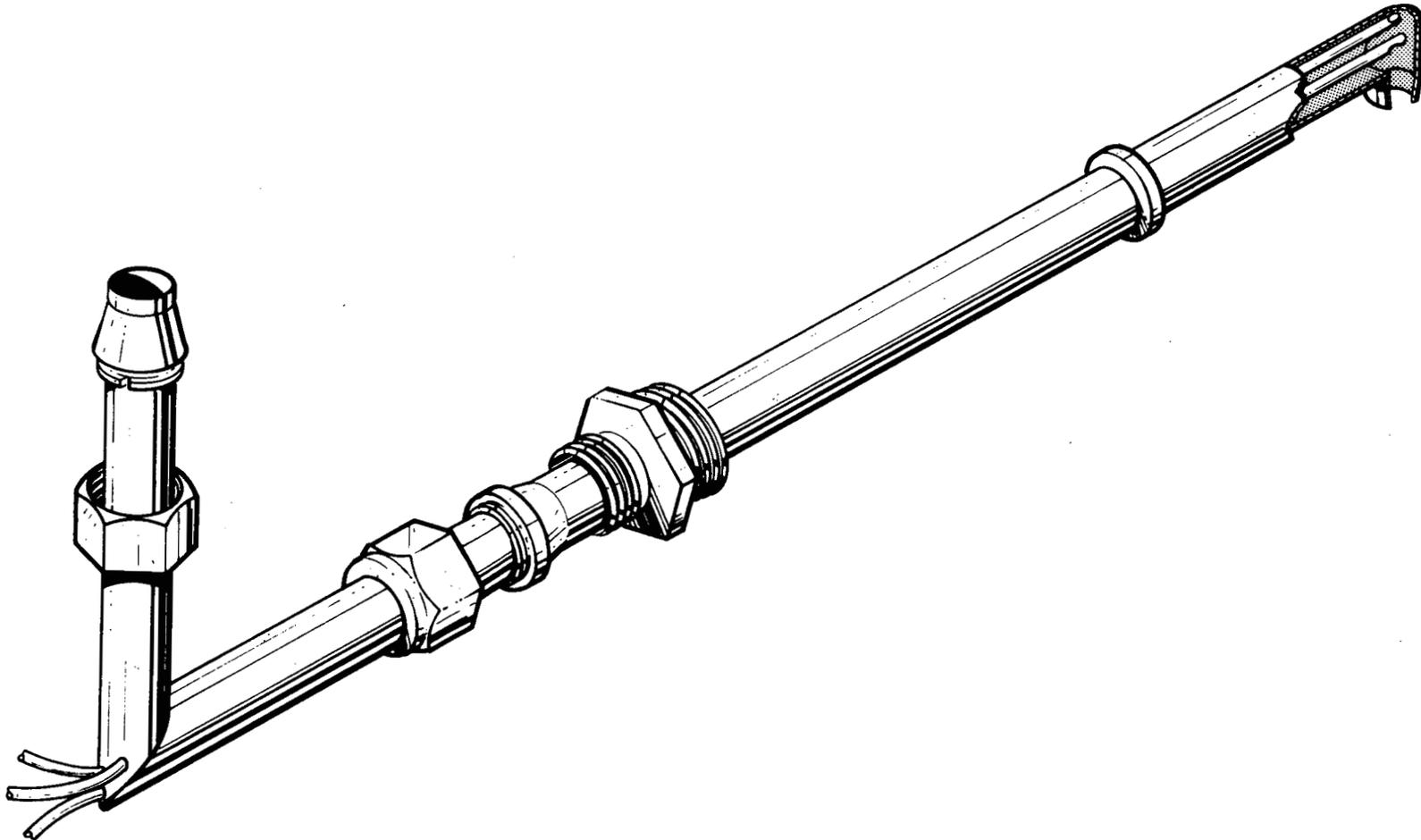


FIGURE 12.1. Isokinetic Sampler

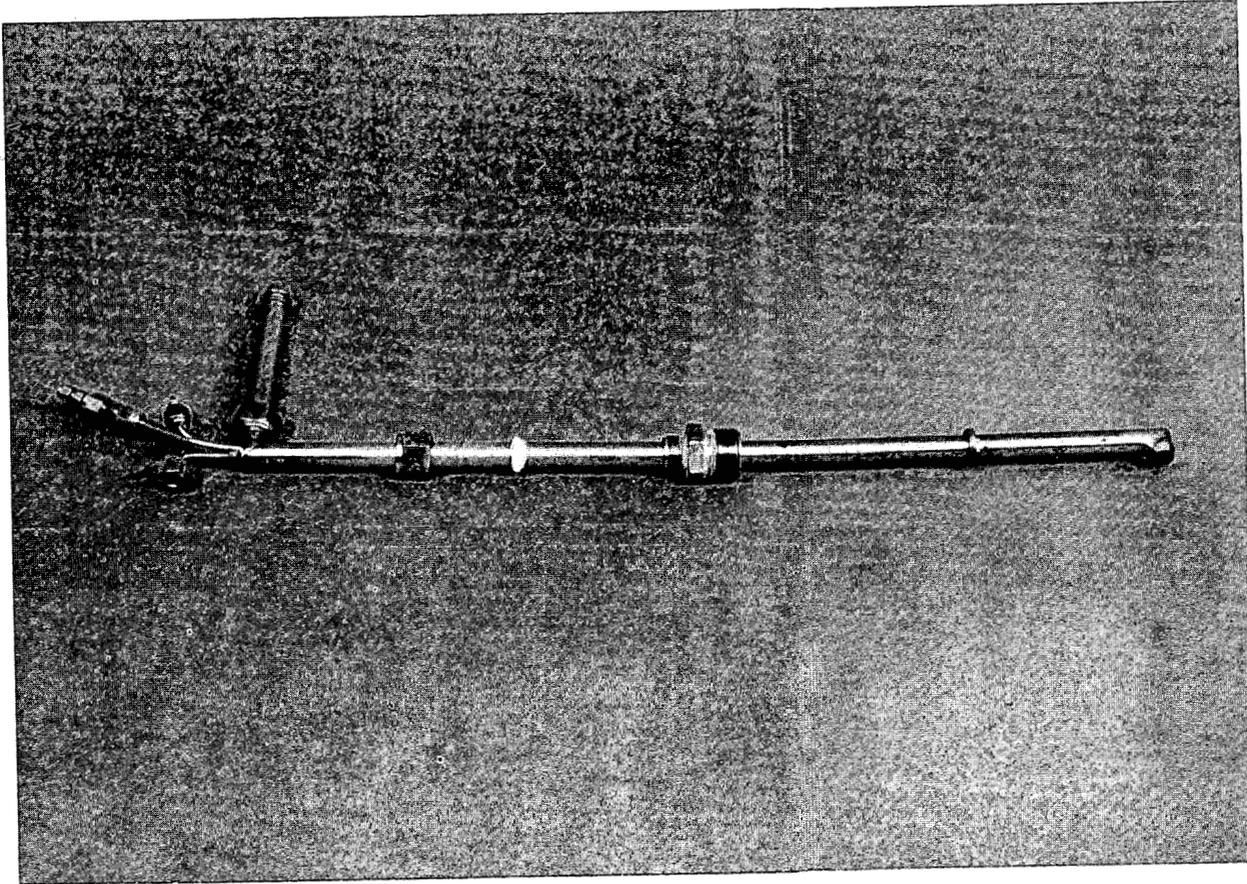


FIGURE 12.2. Isokinetic Sampler

A differential pressure (DP) cell was sized to measure as small a differential pressure as possible between the free stream static pressure and the static pressure; isokinetic flow exists in the sample probe when these pressures are equal. Equipment problems prevented the use of the system as designed and it was not possible to precisely determine the differential pressure.

A major consideration which could not be accommodated within the time available for design was a complimentary heat exchanger system of a suitable size. For the purposes of this first test, a coiled tube immersed in a bucket of cold water was used. This had the disadvantage that during sampling, the temperature of the cooling water rises and a constant temperature differential along the cooling coil could not be maintained. Although the operation in this mode was not steady state, it had the advantages that the total heat transferred from the sample could be measured. Thermocouples, pressure gauges and control valves completed the instrumentation for the sampling system.

#### 12.1.2 Procedure

Reference marks were made on the outside of the probe at 1/2 intervals. The first mark was made with the probe tip located in its lowest position in the pipe and subsequent marks were made along the probe from this point. Prior to start of geothermal fluid flow, the probe was placed at the highest position, i.e., the bottom of the probe was located 4 1/2 inches above the bottom of the 4 inch Sampling Test Loop. This placed the inlet of the probe above the flowing fluid and in a quiet zone in the half coupling welded to the 4 inch pipe.

All connections to the DP cell, thermocouples, other instruments and the cooling coils were completed prior to the start of geothermal fluid flow in the system. When geothermal fluid flow in the Sampling Test Loop reached steady state, sampling was initiated using the isokinetic probe. The isokinetic probe installation is shown in Figures 12.3 and 12.4 and an outline of the sampling system is shown in Figure 12.5.

Samples were obtained at each 1/2 inch interval. The initial sample was taken with the centerline of the I-probe sample port a quarter of an inch below the top of the pipe. Samples were obtained at positions 1 to 8 shown in Figure 12.6 and 12.7. Samples were not obtained in the lowest position, Position 9.

(a)

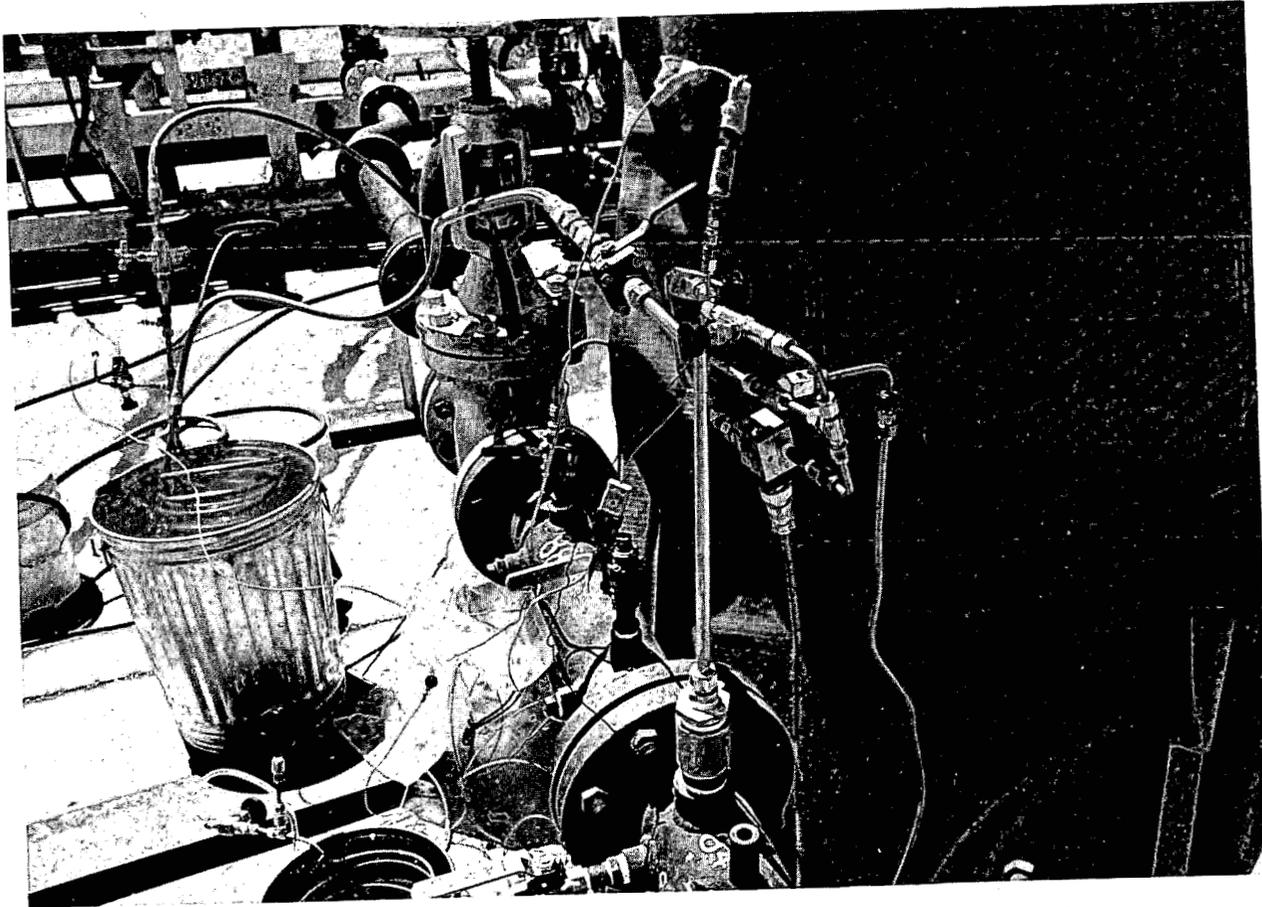


FIGURE 12.3. I-Probe Installation

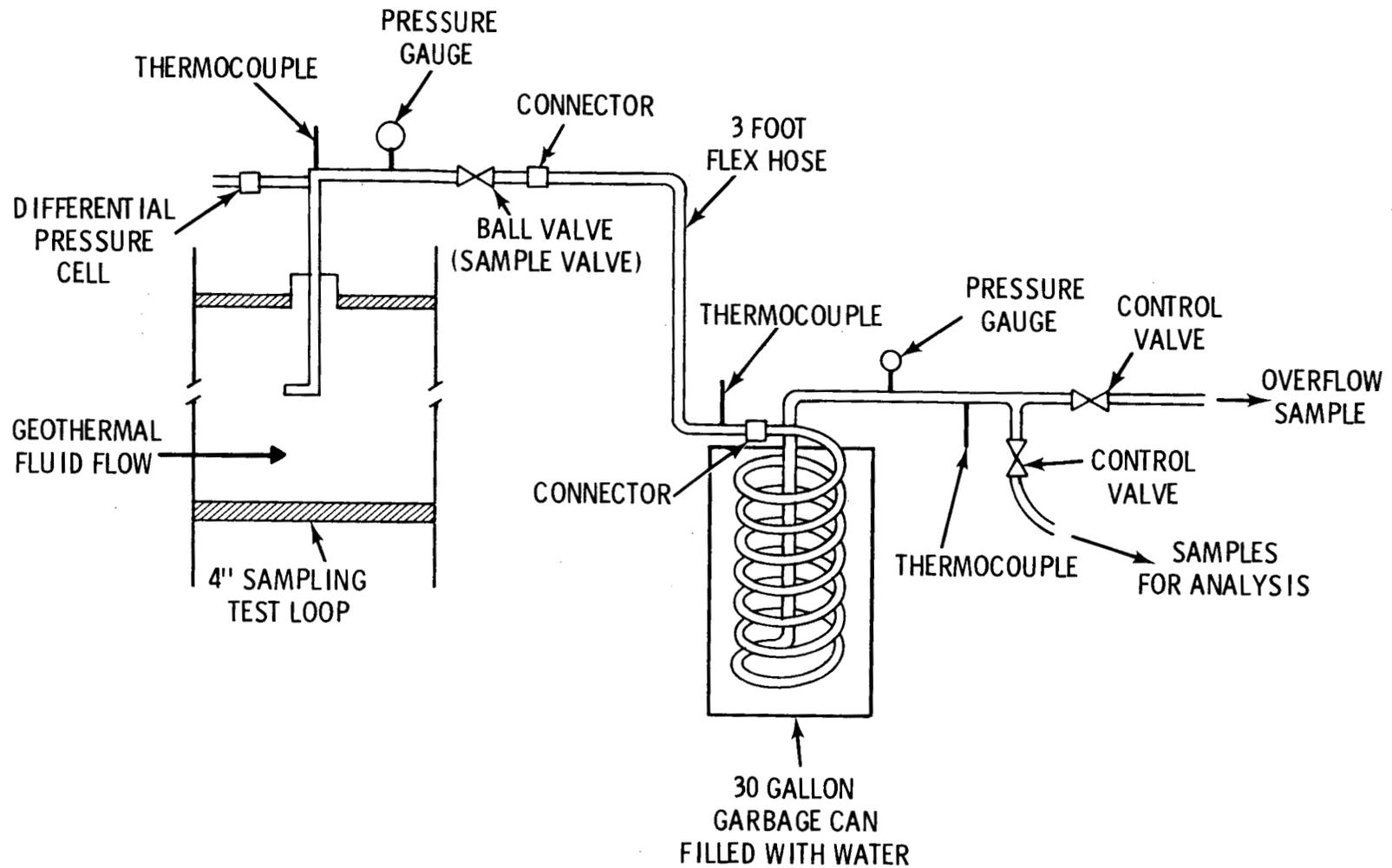


FIGURE 12.5. Schematic of the Sampling System Used in the Isokinetic Test

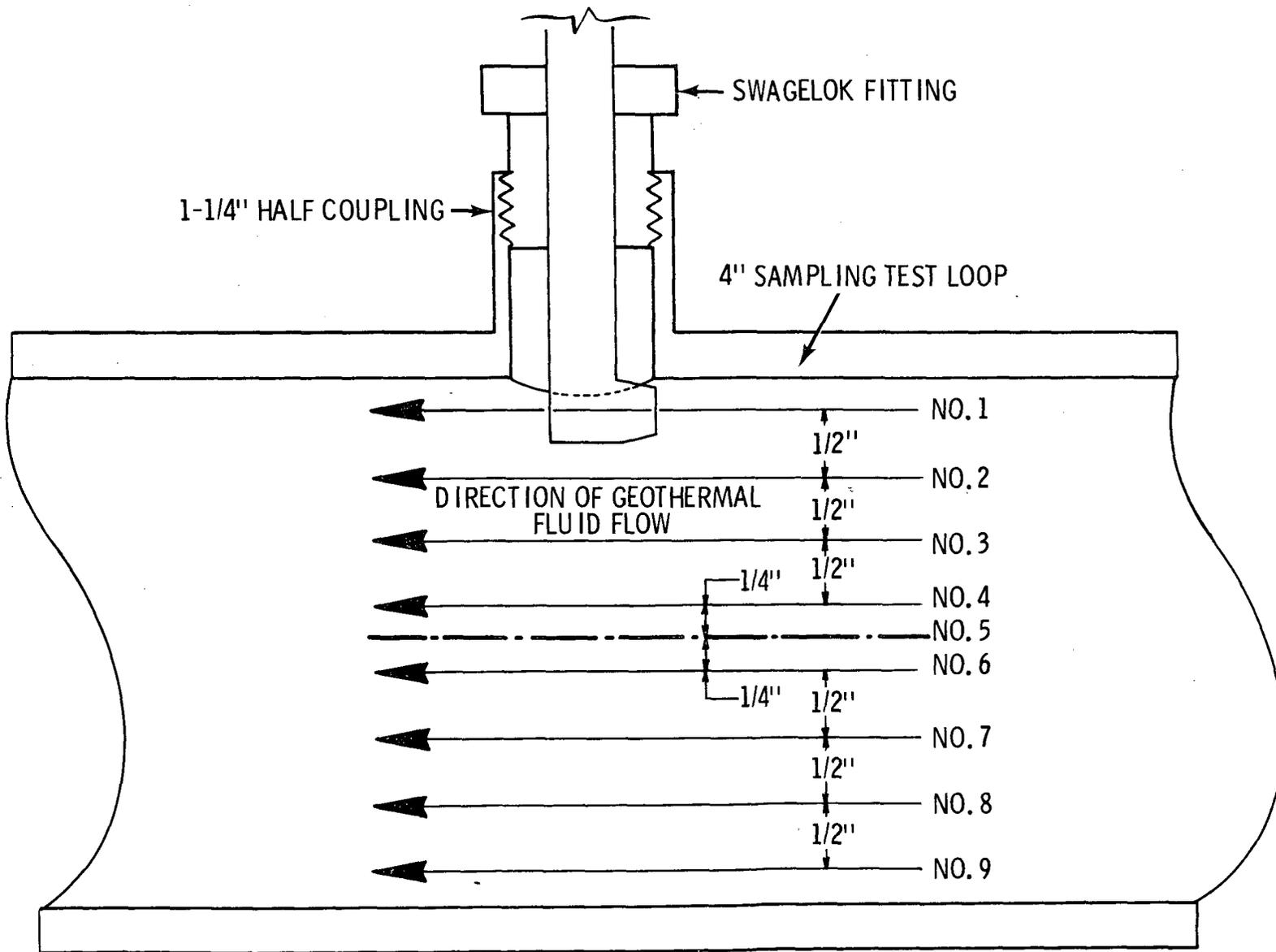


FIGURE 12.6. Probe Positions Where Isokinetic Samples Were Taken

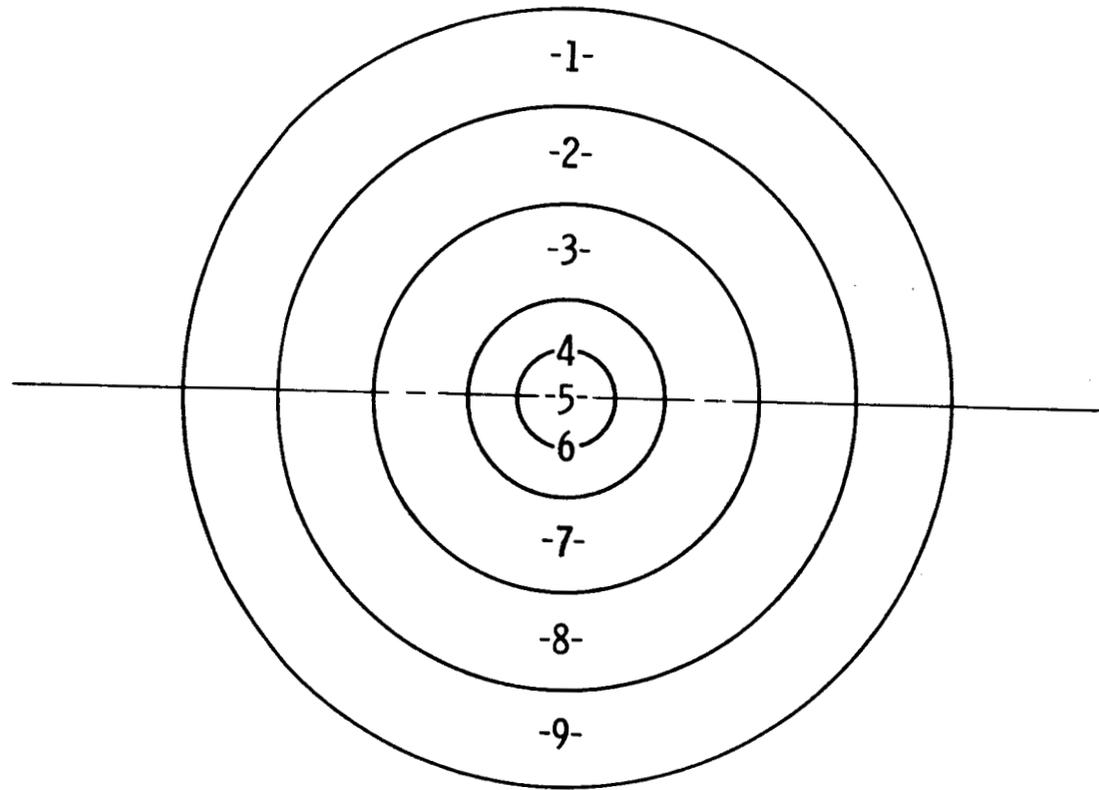


FIGURE 12.7. Location of Sample-Probe Positions

Samples were also taken with the R-probe at the center of the loop after sampling using the I-probe was complete. The port of the R-probe was located approximately 1/2 inch to the right of the I-probe (facing the geothermal fluid flow) during the sampling period.

Samples were obtained as follows. The probe was carefully positioned with the sampling port facing the direction of flow. The probe pressure taps (free stream pressure and geothermal static pressure) were purged with nitrogen to clear the 1/8 inch tubes and both valves to the DP cell were opened. The ball valve upstream of the cooling coil (labeled sampling valve)(Figure 12.5) was opened and the control valve (labeled samples for analysis) was adjusted to cause flow to the sampling apparatus, Figure 12.5. Unfortunately, the sampling apparatus could not handle the total flow from the I-probe and only a small fraction of the total I-probe flow was sampled.

We were also unable to assure that isokinetic conditions existed during sampling due to equipment problems. Since the pressure differential was near zero for nearly all tests, we assume nearly isokinetic conditions existed during sampling. A higher than calculated pressure drop in the cooling coil and problems associated with sampling the total I-probe sample contribute to possible error.

System pressure, the temperature at the probe, I-probe sample temperatures at inlet and outlet of the cooling coil, the temperature of the cooling bath and the bulk liquid temperature of the total I-probe liquid sample were also taken during each run and recorded. The pressure at the outlet of the cooling coil was also obtained during each run and maintained as near zero as possible during sampling to assure free flow in the probe. All of the liquid passing through the I-probe was collected, measured and the average flow rate over the time period of sampling determined. The temperature, pressure and flow rate data thus obtained were sufficient for evaluation of the I-probe. Sampling of the I-probe stream was performed using methods described elsewhere in this report.

### 12.1.3 Results

The data presented here were calculated based on temperature and pressure information available from data logger printouts obtained at the time of sampling log sheets prepared during the test or from chemical analyses, Table 12.1. The data logger sampled the various probes at 5 min intervals and, therefore, much of the needed data were not gathered for these short (5 to 10 min tests).

Samples were analyzed for all elements described in earlier chapters of this report. In addition, the gas/liquid ratio was also determined experimentally on site as was pH, H<sub>2</sub>S concentration and ammonia concentration.

The steam fraction in the fluid was calculated from mass and energy balances using measured compositions, temperature data obtained at the inlet and outlet of the cooling coil and the cold water bath, and average flow rates. Heat loss (or gain) from the bath could be estimated by measuring the temperature drop (or rise) in the bath when the sampler was not operational. The relationships used for this calculation are as follows and their derivations will not be presented here. The data are tabulated in Table 12.1 together with laboratory estimates.

#### Steam Phase Fraction (Based on Chemical Analysis)

$$\frac{G}{L} = \frac{C_b}{C} - 1$$

Assumption: concentration  $C_b$  is constant and equal to the values in Samples MI-84-22/24

#### Heat Input Into Bath

$$Q_1 = m C_p (T_o - T_{ref})$$

#### Average Heat Transfer Coefficient

$$U = \frac{Q_1}{A_o T_{ln}}$$

TABLE 12.1. Sample Data I-Probe Tests

	B-03	MI-13	MI-84							
	23/25, Bulk	01/03, R	01/03, T-1/2	04/06, T-1	07/09, T-1 1/2	10/12, T-2	13/15, T-2 1/2	16/18, T-3	19/21, T-3 1/2	22/24, T-4
pH	6.74	6.49	7.07	6.98	6.72	6.96	6.94	7.40	8.13	8.69
Cond.	6792	6292	2024	2202	5636	5892	6072	7100	6824	6918
SS		62	26	14	11	10	10	10	85	49
HCO <sub>3</sub>	575	--	414.8	430.2	557.2	561.6	569.2	590.2	598.2	605.6
SO <sub>4</sub>	--	164	33.3	41.7	133.3	144.4	148.1	154.1	165.3	166.7
F <sup>-</sup>	3.3	4.4	0.4	0.4	3.0	3.1	3.2	3.6	3.7	3.8
Cl <sup>-</sup>	1917	1952	415	491	1590	1668	1729	1903	2037	2040
NH <sub>3</sub>	14.4	15.8	75.4	--	29.7	26.3	26.2	16.2	8.9	8.4
SiO <sub>2</sub>	201	--	--	--	--	--	--	--	--	--
CO <sub>2</sub>	1155	1615	14719	16545	2694	5189	4554	1988	498	484
Ag	0.38	0.34	0.1	0.1	0.4	0.6	0.5	0.6	0.4	0.5
B	7.79	7.6	1.6	1.9	6.0	6.4	6.5	7.3	7.9	8.0
Ba		0.2	0.4	0.05	0.15	0.17	0.18	0.19	0.24	0.2
Ca	9.4	3.3	1.0	1.6	2.5	3.0	1.5	2.8	3.73	3.9
Fc	0.1	0.1	2.4	2.9	1.2	1.1	1.0	0.7	0.08	0.08
K	103	--	23	27	90	100	107	93	100	--
Li	5.6	5.0	1.05	1.3	3.9	4.3	4.3	4.6	5.2	5.1
Mg	0.3	0.46	0.04	0.23	0.24	0.25	0.19	0.34	0.47	0.5
Ni	1625	1524	325	378	1210	1282	1292	1440	1605	1605
P	--	--	0.1	0.5	0.5	--	0.5	0.5	--	--
Si	1049	102.8	22	25.9	82.3	87.5	89.2	99.4	109	109
Sr	2.25	0.71	0.2	0.2	0.68	0.7	0.69	0.78	1.0	0.9
G/L	--	0.12	--	0.91	0.20	0.59	0.64	0.15	0.02	0.02

GL = Gas-Liquid Ratio

$$T_{lm} = \frac{(T_i - T_o)}{\ln \frac{(T_i - T_o)}{(T_o - T_b)}}$$

Sensible Heat Lost By Brine

$$Q_2 = [(CpTW)_{iw} + (CpTW)_{is} - (CpTW)_o] \tau$$

$$W_{is} \ll W_{iw}, W_{iw} \sim W_o \approx W$$

$$Q_2 \approx (WCpT_i - WCpT_o) \tau$$

Heat of Condensation

$$Q_v = Q_1 - Q_2$$

$$\frac{G}{L} = \frac{Q_v}{\frac{\lambda}{W\tau}}$$

Nomenclature

C = Concentration, mg/l

C<sub>b</sub> = Concentration in MI-84-22/24

G = Gas Flow Rate, lb/min

L = Liquid Flow Rate, lb/min

m = Mass, lb

Cp = Heat Capacity,  $\frac{Btu}{^{\circ}F \text{ lb}}$

T = Temperature,  $^{\circ}F$

T<sub>lm</sub> = Log Mean Temperature Difference,  $^{\circ}F$

A<sub>o</sub> = Heat Transfer Surface Area, 2.814 ft<sup>2</sup>

U = Heat Transfer Coefficient,  $\frac{BTU}{hr \text{ ft}^2 \text{ } ^{\circ}F}$

$^{\circ}F$  = Fahrenheit Degrees

Q<sub>1</sub> = Heat into Tank, BTU

Q<sub>2</sub> = Heat Lost by Brine, BTU

ρ = Density, Mass Per Unit Volume

τ = Time, min

$\lambda$  = Latent Heat of Vaporization, BTU/lb

W = Brine Flow Rate, lb/min

Subscripts:

i = At Heat Exchanger Inlet

o = At Heat Exchanger Outlet

w = Water

s = Steam

Steam fraction, gas/liquid ratio, pH, conductivity, and elemental or chemical composition at the different locations sampled followed the trends expected for the experiment. These results are plotted in Figures 12.8 to 12.17.

Steam and ammonia content is high in the upper section of the sample test loop, drops sharply as the centerline is approached and is nearly zero in the lower 2 1/4 to 2 1/2 inches of the pipe. This is expected since these are volatile components. Corresponding but inverted results were observed for suspended solids, pH and elemental analyses which are indicative of nonvolatile species. Here, lower concentrations of ionic species were observed in the upper section of the pipe and increased sharply as the I-probe entered the liquid phase. pH and suspended solids increased slightly near the top of the pipe probably because condensed steam driven along the upper pipe surface entered the isokinetic sample port. Thus, the change in composition is not as sharp since the liquid is more dilute than "pure" brine.

The gas/liquid ratio correlation shows the expected trend; however, major discrepancies are observed in the data at Sample Locations 3 and 4 just above the centerline of the pipe. An interfacial region of high turbulence between gas and liquid flow was expected in this region. In addition, we were not able to sample the whole stream passing through the I-probe but had to take a sidestream for samples. Where major flow through the I-probe was either gas or liquid, the effects of taking a sidestream would be minimal. In the interfacial mid-region of the Sample Test Loop near the gas/liquid interface, gas contained in the fluid passing through the I-probe would take the path of least resistance. This could bias the results in favor of a lower gas/liquid ratio at Positions 3 and 4.

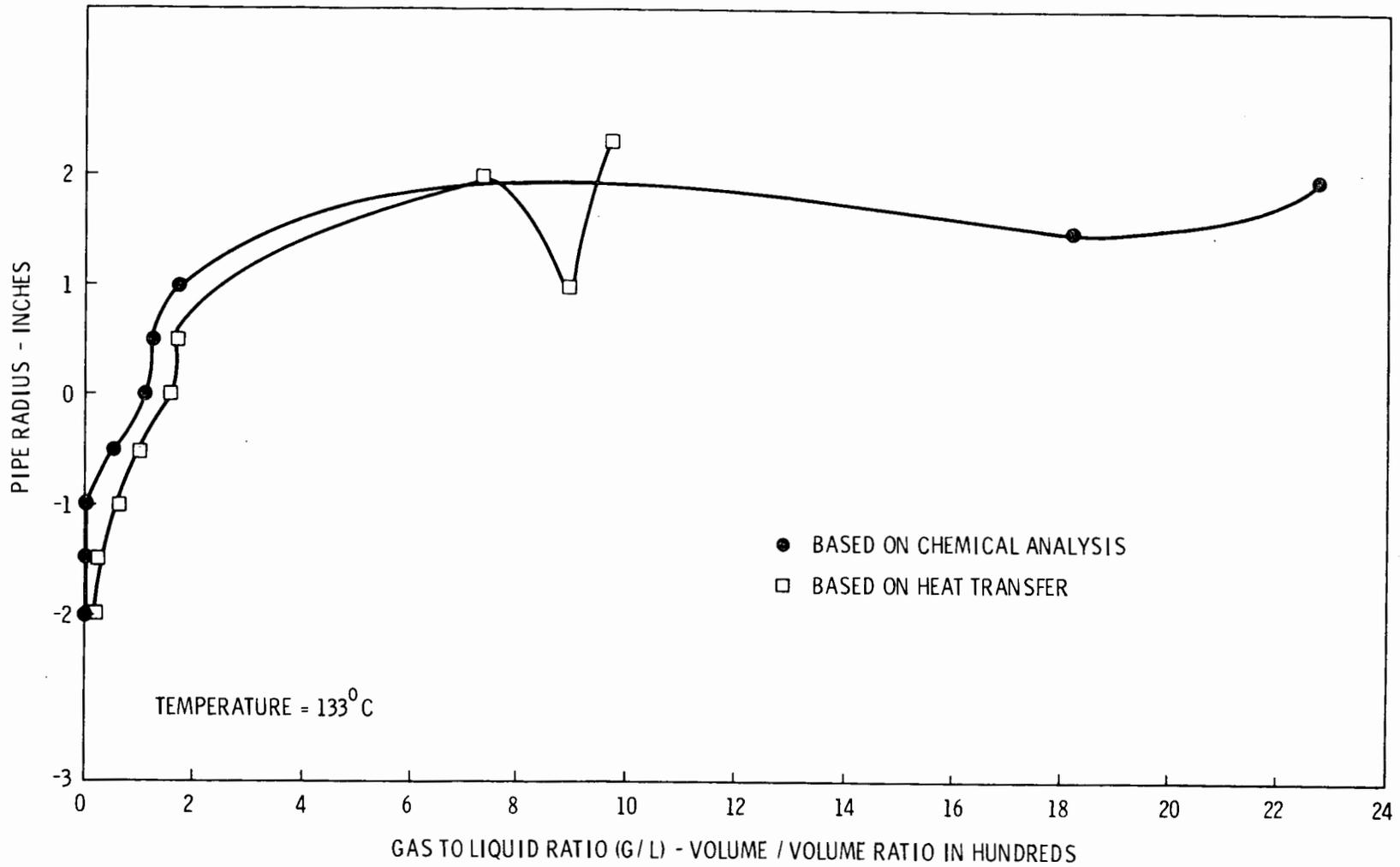


FIGURE 12.8.

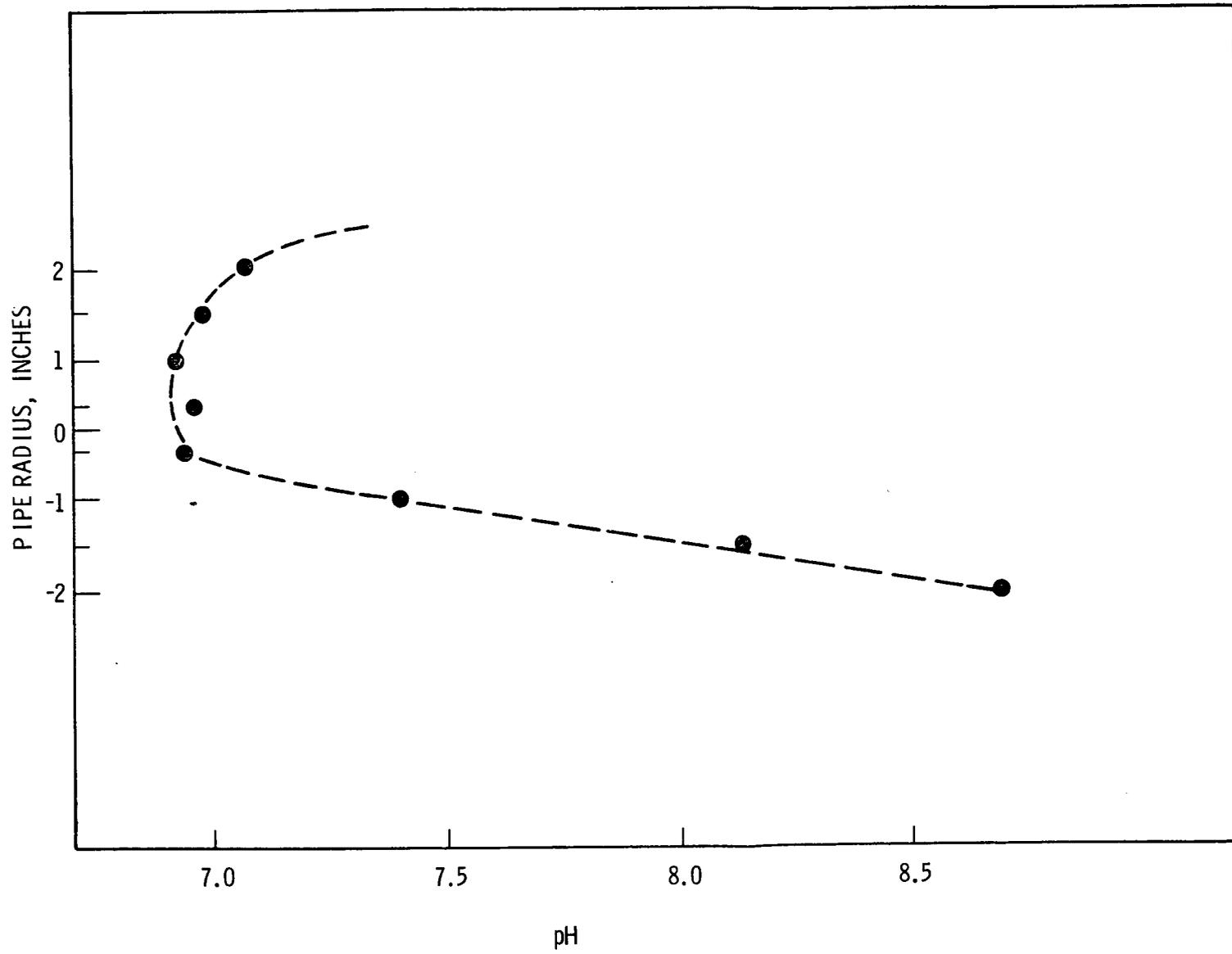


FIGURE 12.9. Vertical Distribution of pH

12.17

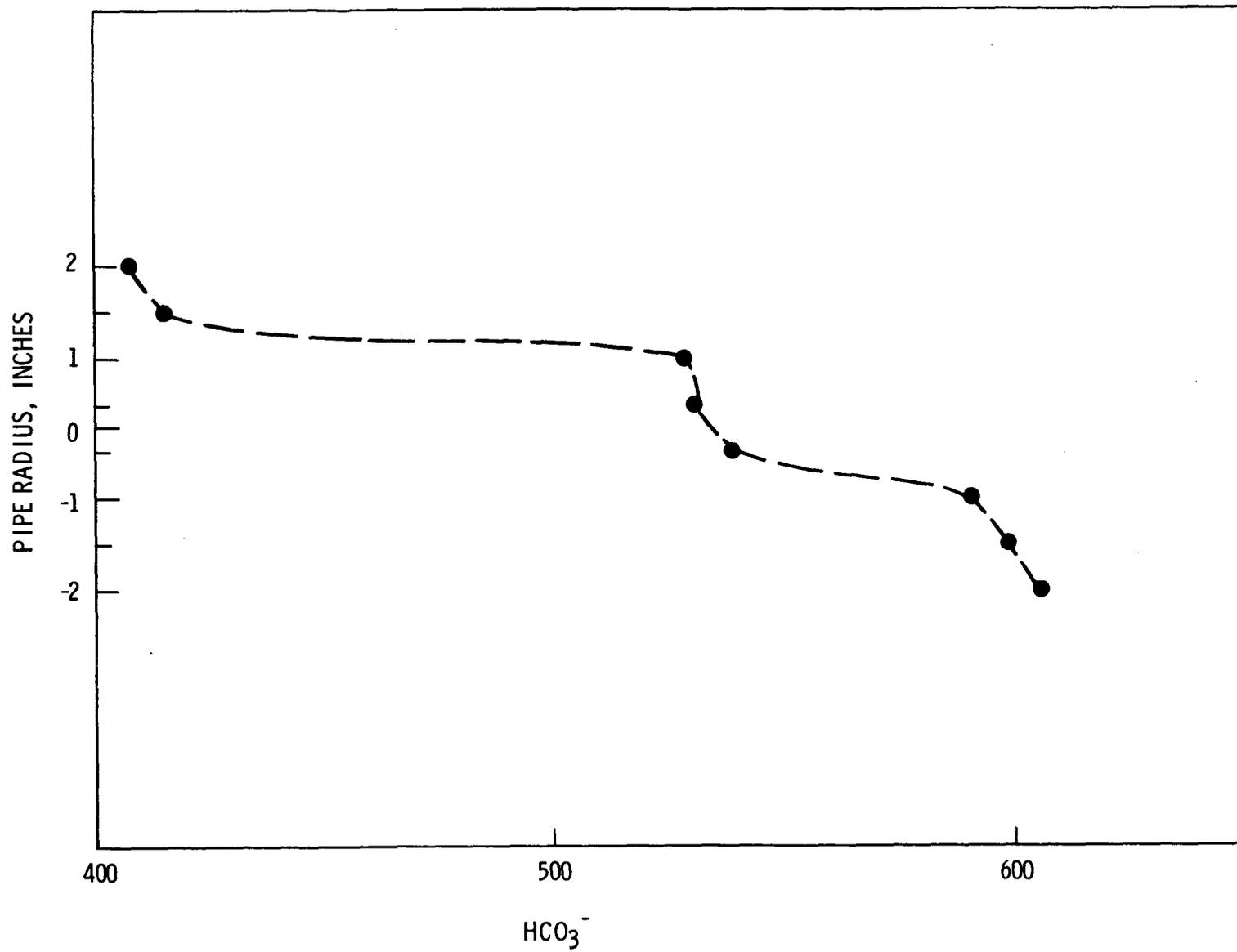


FIGURE 12.10. Vertical Distribution of Bicarbonate

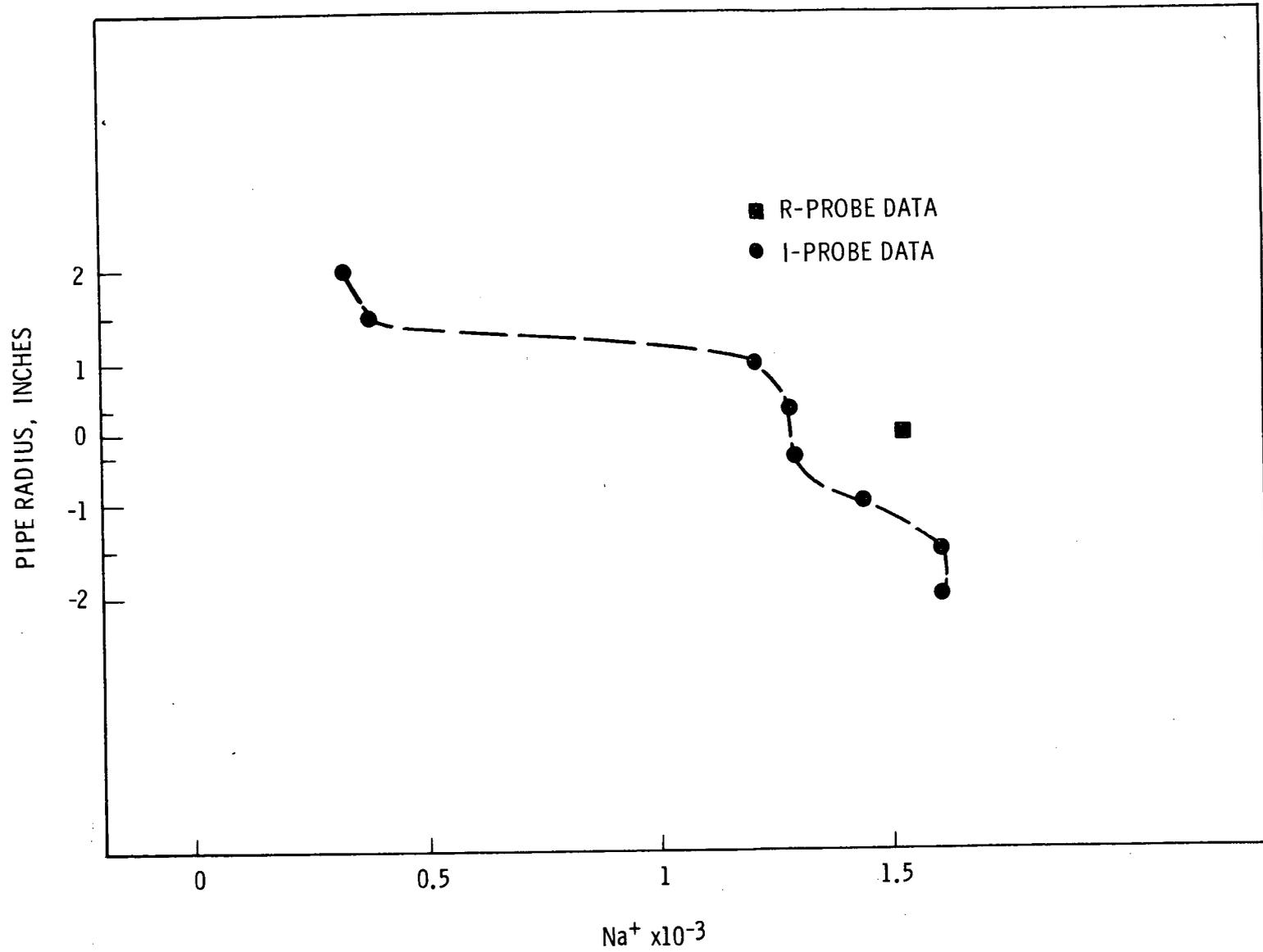


FIGURE 12.11. Vertical Distribution of Sodium

12.19

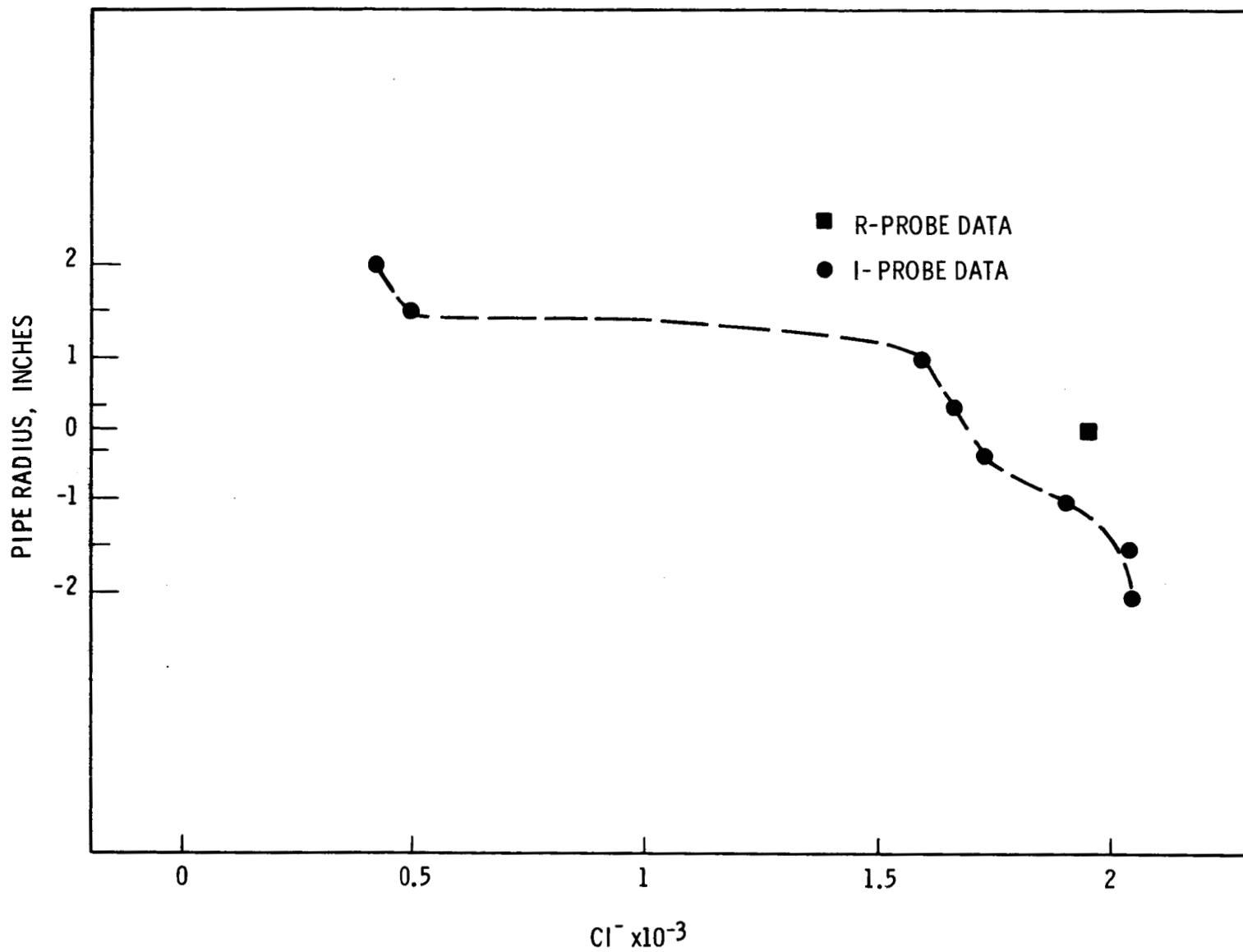


FIGURE 12.12. Vertical Distribution of Chloride

12.20

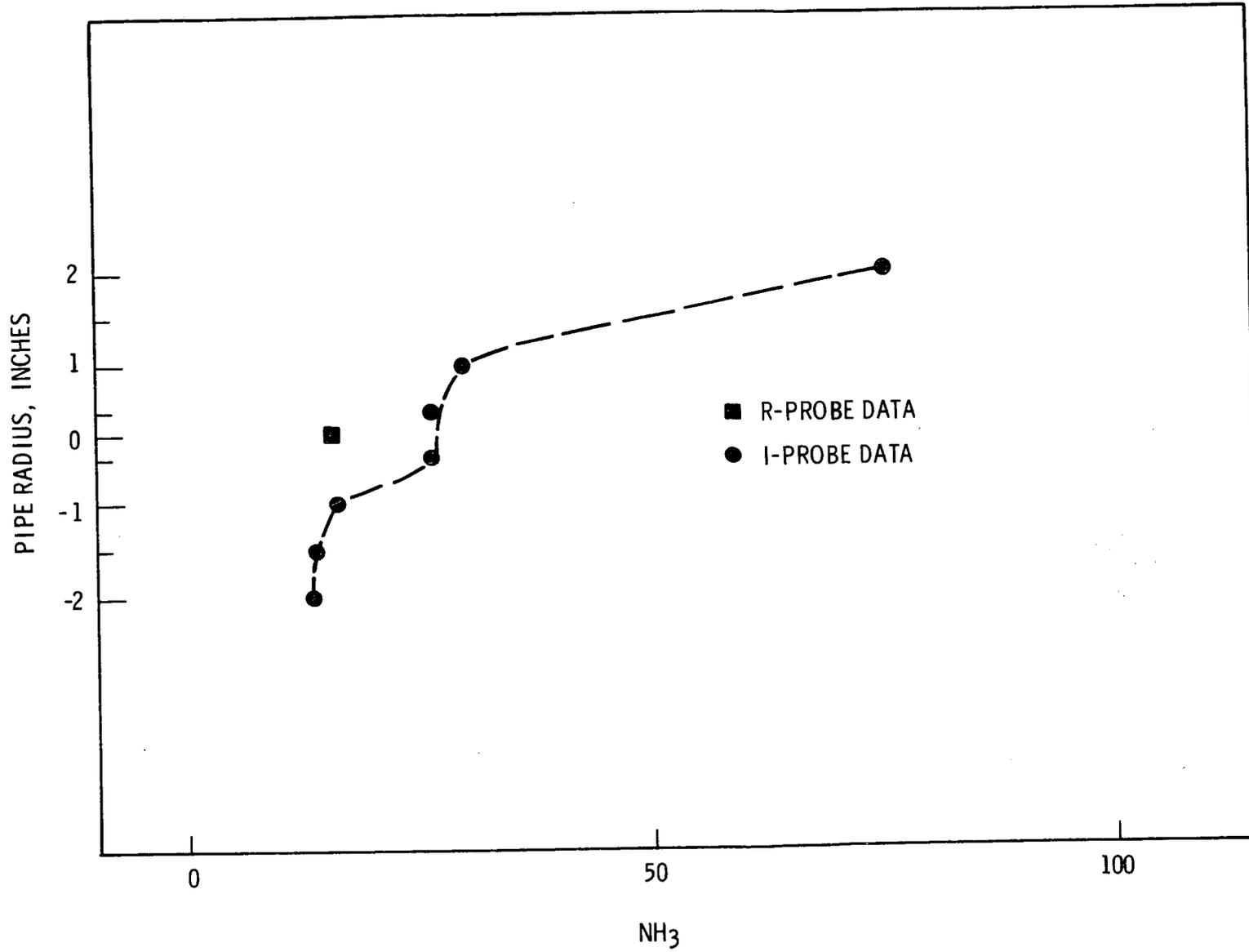


FIGURE 12.13. Vertical Distribution  $\text{NH}_3$

12.21

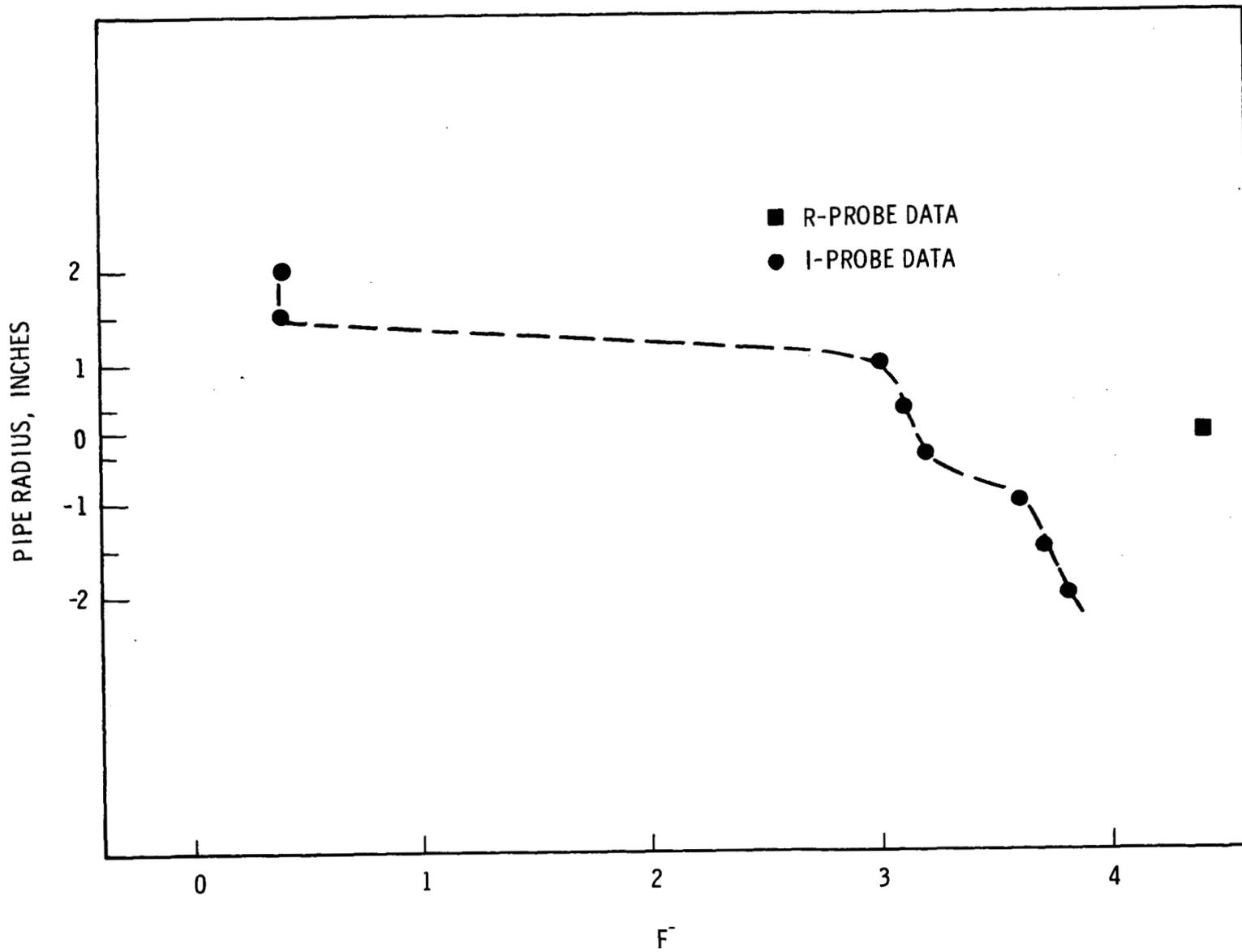


FIGURE 12.14. Vertical Distribution of Fluoride

12.22

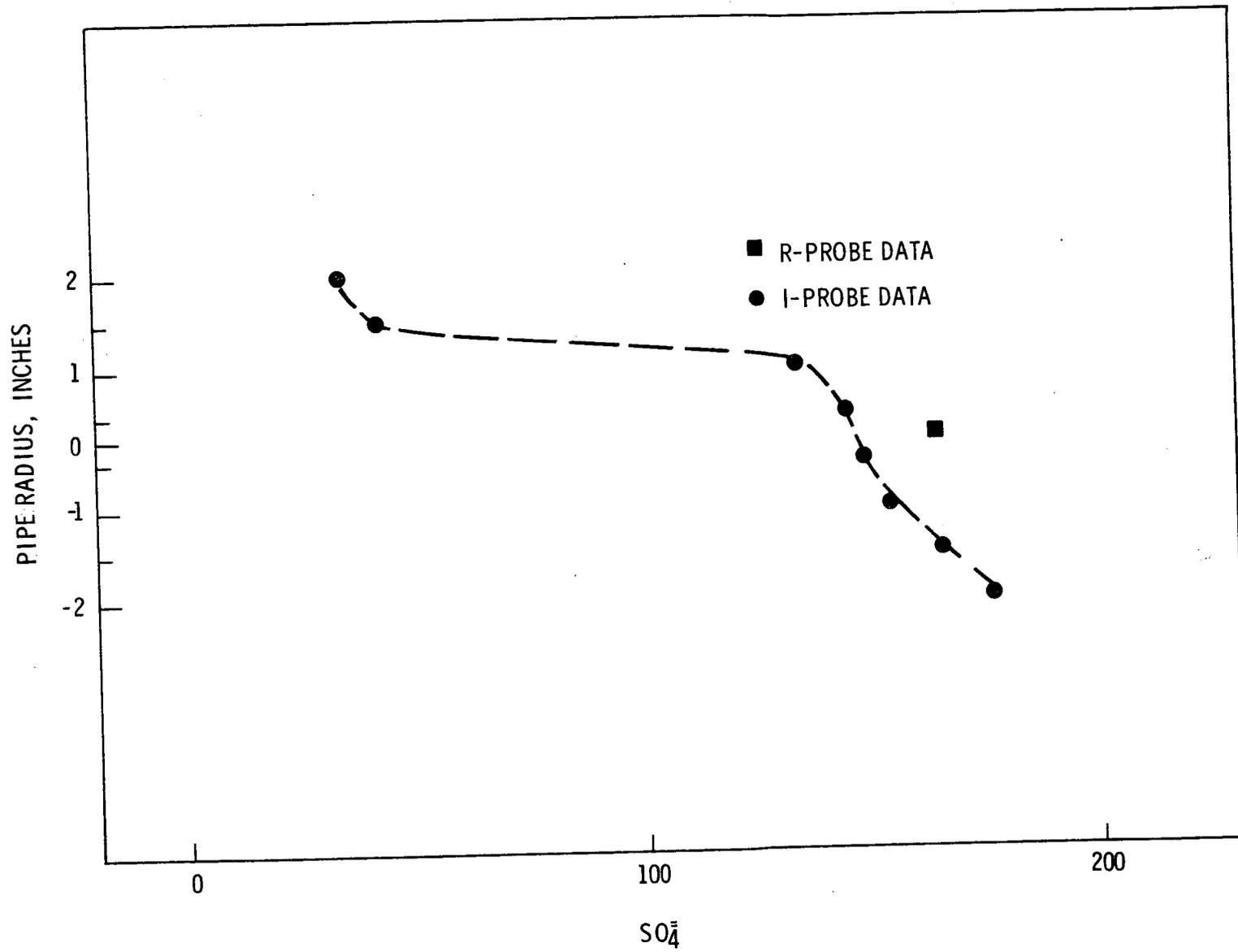


FIGURE 12.15. Vertical Distribution of Sulfate

12.23

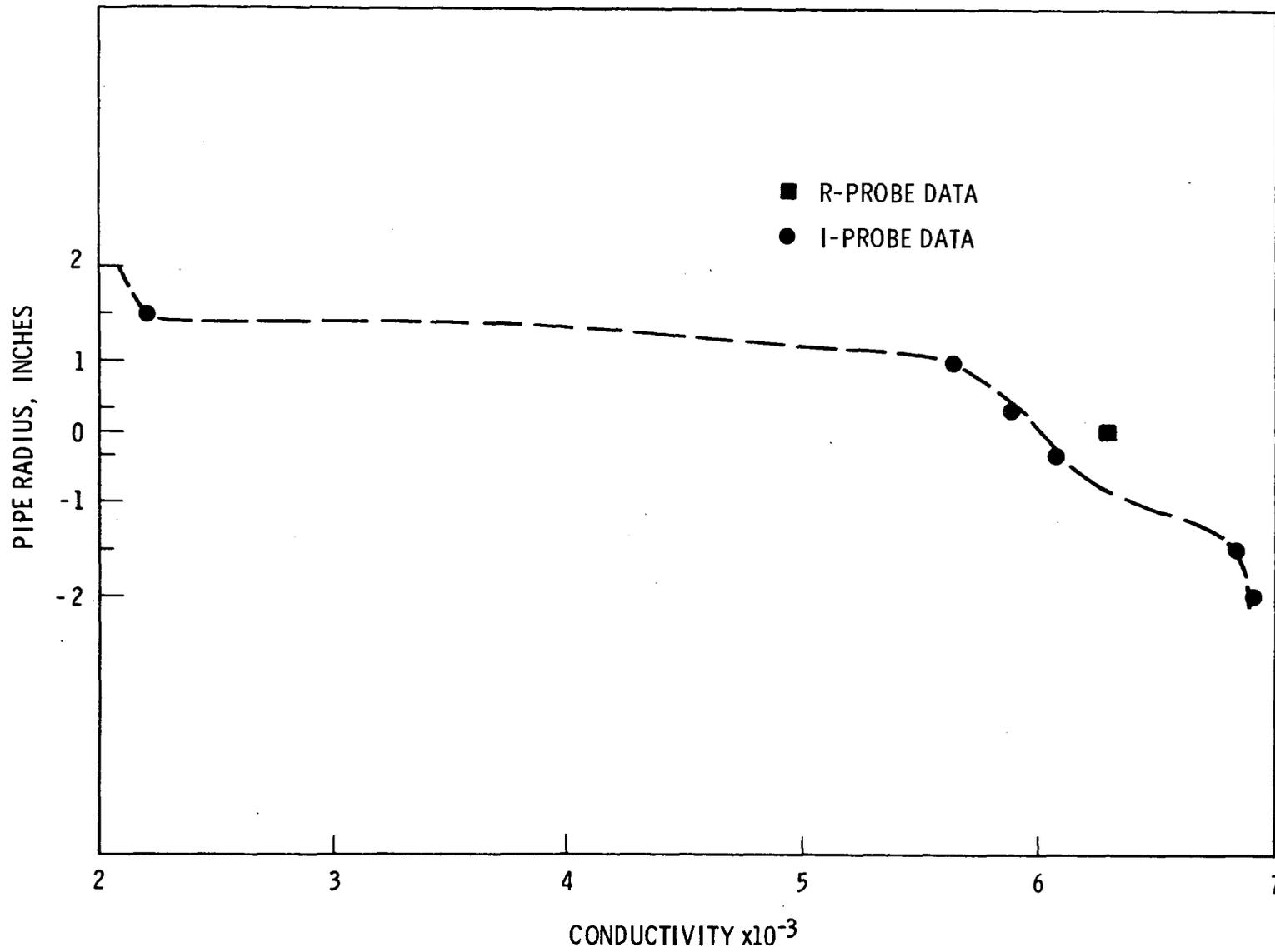


FIGURE 12.16. Vertical Distribution of Conductivity

12.24

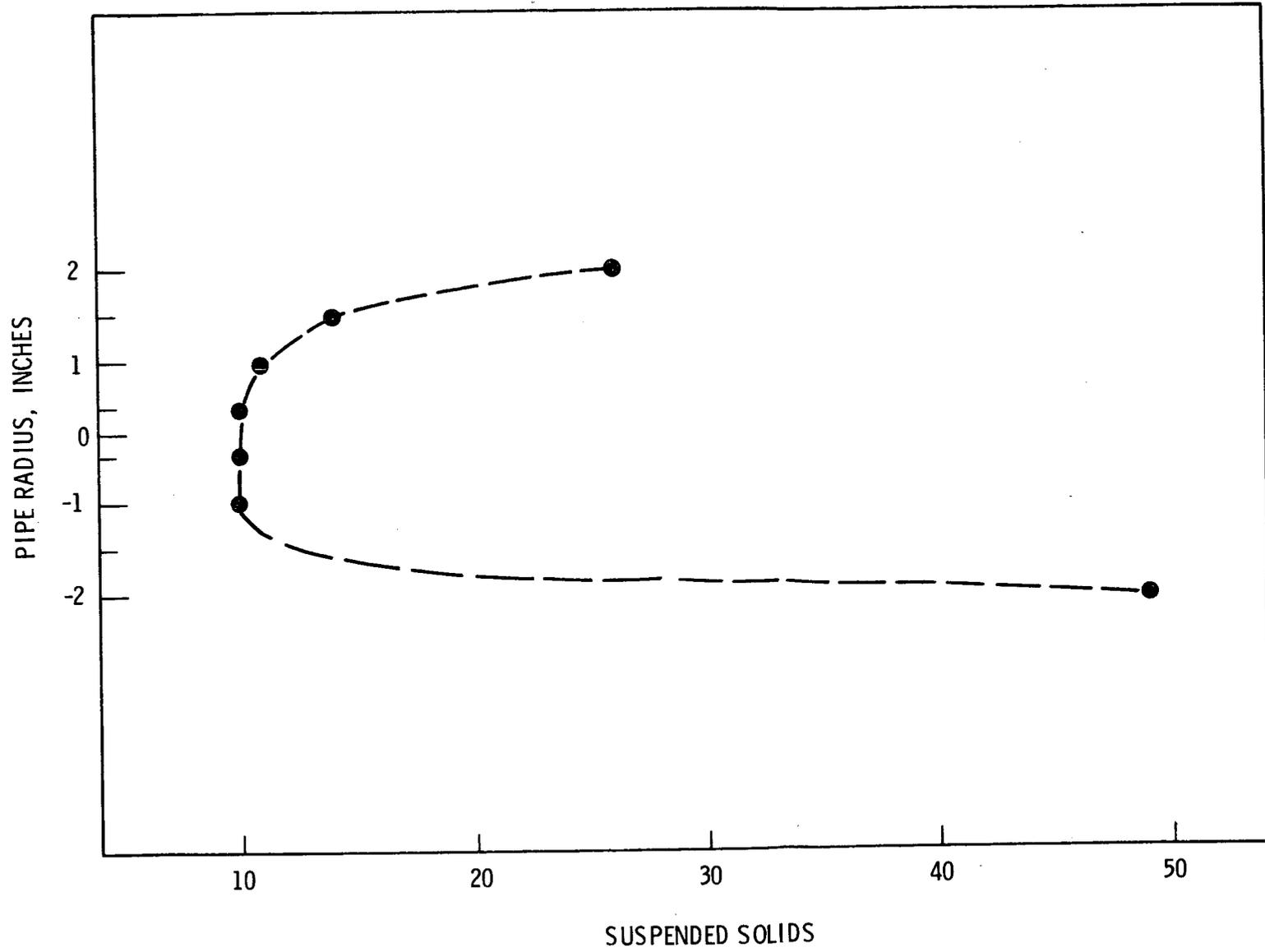


FIGURE 12.17. Vertical Distribution of Suspended Solids

The G/L ratios presented in Table 12.2 shows distinct differences. This is because:

- The "lab values" are the total noncondensable gas to total liquid (including steam) ratios referenced to 34°C. The remaining two sets of data are actually steam to liquid ratios, i.e., no consideration is made for entrained CO<sub>2</sub>. Further, they are referenced to 133°C, the operating temperature.
- The data by the "chemical analysis" and "heat transfer" methods differ from one another at the high and low positions in the pipe. Errors in the low positions are due to the short test runs at the high liquid flow rates and the shortage of good heat transfer data.

TABLE 12.2. Results of Gas to Liquid Ratio Calculations

<u>I-Probe Position</u>	<u>Flow Rate (<math>\ell</math>/min)</u>	<u>G/L (a) (Lab Value)</u>	<u>G/L (b) (Chem Anal)</u>	<u>G/L (c) (Heat Transfer)</u>
1	0.68	NA	2275	730
2	0.78	0.91	1820	890
3	1.4	0.2	170	165
4	1.73	0.59	125	155
5	1.77	0.64	110	100
6	2.69	0.15	55	59
7	5.36	0.02	2	18
8	5.30	0.02	0	18
9	NT	NT	NT	NT

NA = Not Available

NT = Not Taken

(a) Field value: milliliters of noncondensibles to milliliter of brine plus condensibles at 34°C, supplied with lab data.

(b) Based on ionic compositions of samples; adjusted to 133°C; milliliter condensibles to milliliters brine, does not include noncondensibles.

(c) Based on heat transfer data; otherwise same as b.

Near the top of the pipe, steam condensate flowing along the pipe wall may have entered the probe to give erroneous values. This hypothesis is backed by apparent increase in the amount of steam from the "chemical analysis" data and an apparent decrease from the heat transfer data.

Thus, at the bottom of the pipe, the "chemical analysis" data are thought to be more valid and at the top, the heat transfer data. In the center, they are in agreement.

Separation of  $\text{CO}_2$  from the geothermal fluid appears to be complete by the time the fluid reached the I-probe because  $\text{HCO}_3^-$  concentration did not change drastically at locations deeper in the liquid phase and  $\text{CO}_2$  outgassing was not obvious in the liquid samples from Positions 6, 7 and 8.

Based on the compositions measured in the liquid samples<sup>(a)</sup> and the liquid flow rates (Table 12.2), the composition of the bulk brine was calculated, Table 12.3. These calculated values were compared to bulk values at Test Point 3. For comparison, bulk brine values reported in Table 11.5 and local values (Point 84) measured with the R-probe are included.

In general, the measured and calculated data agree within 10%. The "elements" which differ significantly from 10% are: flow rate, total  $\text{CO}_2$ , As, Ba, Ca, Fe, Li, Mg, and Sr. In all cases, particularly in the case of flow rate, more detailed information on the flow pattern is needed. A pattern for obtaining these data is shown in Figure 12.8; detailed information could be obtained with additional traverses of the pipe, in the horizontal and  $45^\circ$  directions. As a minimum, the horizontal traverse should be made in addition to the vertical traverse. Obviously, a very large number of samples are required to characterize the flow.

The Fe data are dismissed because the data show an increase of Fe level in the vapor. Because the samples were taken, chronologically, from the top down, we suspect corrosion (or cleaning) of the sample line; all parts of which were being used for the first time. Moving the probe upward during sampling was not possible.

---

(a) No samples were taken in the bottom "half" inch, Point 9, of the pipe. All data were assumed to be about the same as the "one inch" above the bottom point, Point 8. This was assumed due to the similarity of Points 7 and 8.

TABLE 12.3. Comparisons of Bulk Properties

	A	B	C	D	$\frac{D-C}{C} \times 100$
	Reported in Table 11.5	R-Probe Data Single Position	Reported in Data Sheets Avg of B-02-23/25	Calculated From "Isokinetic" Data	(%)
Flow (a)	--	--	85 (b)	110	+30
pH	5.8	6.49	6.74	7.69	--
Cond. (c)	--	6292	6792	6394	-6
HCO <sub>3</sub> <sup>-</sup> (d)	--	589	575	583	+1
SO <sub>4</sub> <sup>-</sup> (d)	--	164	--	153	--
F <sup>-</sup> (d)	--	4.4	3.3	3.4	+3
Cl <sup>-</sup> (d)	1901	1952	1917	1869	-3
NH <sub>3</sub> (d)	--	15.8	14.4	15.0	+4
Total CO <sub>2</sub> (d)	1155	--	1155	1672	+45
As (d)	0.7	0.34	0.38	0.45	+18
B (d)	7.5	7.6	7.7	7.3	-6
Ba (d)	0.3	0.2	0.3	0.21	-30
Ca (d)	8.5	3.3	9.4	3.5	-62
Fe (d)	0.2	0.1	0.1	0.4	+300
K (d)	--	--	103	93	-10
Li (d)	4.8	5.0	5.6	4.7	-16
Mg (d)	0.2	0.46	0.3	0.44	+47
Na (d)	1512	1524	1625	1466	-10
Si (d)	101	102.8	104.9	100	-4
Sr (d)	2.1	0.71	2.25	0.88	-61

(a) Gal/min.

(b) Based on Fluke data logger tapes.

(c) mho/cm.

(d) mg/l.

The Ca and Sr are expected to deposit as scale and are expected to be in lower concentration than the true bulk brine. The As and Mg are such small numbers that a large error is not unexpected.

One possible explanation for the high total CO<sub>2</sub> value is the isokinetic sample stream was itself sampled, non-isokinetically. Conceivably this might have increased the relative amount of CO<sub>2</sub> collected.

## 12.2 CONCLUSIONS

The above results demonstrate that the isokinetic sampling probe is a useful diagnostic tool for defining the flow pattern and sampling geothermal fluids flowing in piping systems. Representative two-phase samples at the sample probe tip can be obtained for analysis in dilute two-phase streams.<sup>(a)</sup> If sufficient samples using different traverses of the flow stream are obtained an approximation of the bulk flow composition of the soluble salts can be calculated. However, more work and testing of isokinetic probes is needed to show how "true" compositions of the supply well can be calculated. In order to completely characterize the geothermal reservoir chemistry, a mini research project is required to take all the analytical data and calculate a "composite" composition.

A better calorimeter is required than the coil-in-bucket that was used here. The two best candidates are a single pass counter flow heat exchanger and a regenerative heat exchanger.

If further experiments are undertaken, the sampling apparatus should be designed to sample the total flow passing the isokinetic probe rather than taking a small portion of the flow. In addition, the pressure measuring system used to determine the isokinetic point needs to be improved to provide more assurance of isokinetic conditions. Additional samples should be taken at different angles across the centerline of the pipe to establish the liquid-gas profile.

---

(a) A dilute phase is where a major portion of the phase is either gas or liquid and the diluent is in the form of droplets, bubbles or solid particles. Where a stream contains significant fractions of both gases or liquids, uncertainties increase and data obtained from the samples must be interpreted with great care.

Until two-phase sampling is proven, we recommend the well chemistry be established by directing the flow into a full size steam/liquid separator and using single phase sampling as shown in Figure 11.10.

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## 13.0 SINGLE-PHASE SAMPLING PROCEDURE FOR GEOTHERMAL SYSTEMS

### 13.1 INTRODUCTION

#### 13.1.1 Purpose

This procedure permits the practiced operator to sample single-phase flows to determine chemical and physical characteristics of the fluid. This method and appropriate supporting analytical techniques will provide the following data:

1. Liquid
  - Elemental Composition
  - pH
  - Redox Potential
2. Gases
  - Amount
  - Chemical Composition
3. Gas/Liquid Ratio at Reduced Pressure
4. Solids
  - Composition
  - Abundance in Flow

#### 13.1.2 Safety

Two major safety concerns are high temperature and high pressure. The high temperatures can be guarded against by wearing insulated gloves. The danger from high pressure can be reduced by wearing safety glasses and by mechanically restraining the sample probe to prevent expulsion under pressure.

#### 13.1.3 Special Concerns

The sample containers should be prewashed to prevent contamination.

#### 13.1.4 Calculations

The only result that can be mathematically calculated directly from the field data is the gas:liquid ratio. Other parameters are either determined directly from instruments in the field or calculated based on subsequent laboratory analysis.

#### 13.1.5 Abbreviations

FU = Filtered, Unacidified Fluid  
FA = Filtered, Acidified Fluid  
RU = Raw, Unacidified Fluid (Unfiltered)  
DI = Deionized  
ZnAc = Zinc Acetate

### 13.2 EQUIPMENT

#### 13.2.1 Sampling Line

1. Insertion sampling probe (1/4" diameter) with flex hose and twin cooling coils.
2. Two 5-gallon buckets (for cooling baths).
3. Inlet and outlet valve and temperature components with suitable pipe fittings to attach to system (see drawing).
4. Sample containers:
  - 3 each 1 liter bottles for FU, FA, and RU samples (FA bottle to contain 10 ml HCl - Ultrex<sup>®</sup> brand by J. T. Baker)
  - 1 each 100 ml bottle (for SiO<sub>2</sub> sample dilution)
  - 1 each 500 ml bottle for CO<sub>2</sub> containing 250 ml (2 N) NaOH (for CO<sub>2</sub> stabilization)
  - 1 each 1 liter bottle containing 500 ml (0.5 N) ZnAc (for H<sub>2</sub>S)
5. Sparge tube and stopper.

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<sup>®</sup> J. T. Baker Chemical Company

6. 1 500 ml gas bulb.
7. 2 plastic pails and bailing cup to remove excesses from cooling buckets and sample line.
8. Heat insulating gloves.
9. Safety glasses.
10. Ice (plenty).
11. Water (plenty).
12. Glass separator in stand (gas/liquid ratio).
13. 1/4" Tygon<sup>®</sup> tubing and pinch clamp (gas/liquid ratio).

#### 13.2.2 Measurement Tools

1. pH meter and probe and buffer solutions. If in-line probe is used, verify that probe and housing will withstand full system pressure; if not, delete in-line cell.
2. 2 ml pipette and 100 ml volumetric flask for SiO<sub>2</sub> dilution.
3. 1 liter DI water in squeeze bottle.
4. 1,000 or 2,000 ml graduated cylinder.
5. Barometer.
6. 1 each kits (Chemetrics)<sup>®\*</sup> for field determination of O<sub>2</sub>, H<sub>2</sub>S, NH<sub>3</sub>.
7. Clip board with data sheet.
8. Thermometer for water and air temperature.
9. Redox probe and meter (if requested).
10. Wet test cumulative flow meter (filled with DI water to top of sight glass pointer) to measure gas flow for gas:liquid ratio.
11. Stopwatch - gas:liquid ratio.
12. 1 preweighed filter (millipore) in protective holder - tared to 10<sup>-4</sup> grams for suspended solids.

---

\* A typical product is from Chemetrics Inc., Warrington, VA.

### 13.3 PROCEDURE

#### 13.3.1 Start

1. Obtain well flow data to assure normal conditions.
2. Get permission from well operator before sampling any well or plant system.

#### 13.3.2 Sample Train Assembly

1. Assemble equipment and connect sample train components. PUT ON SAFETY GLASSES!
2. Attach insertion probe to flex hose with probe valve closed.
3. Attach ball valve/pressure gland to system valve. (Note: if sampling plant inlet or outlet test spools, attach flex hose directly to sample valve without using probe/gland.)
4. Calibrate pH probe in buffers.
5. Check all valves to assure they are closed.
6. Connect cooling coils to flex hose and insert probe to center of loop pipe as follows:
  - (a) Measure distance probe is to be inserted and mark probe.
  - (b) Insert probe into pressure gland and tighten - be sure safety chain is attached.
  - (c) Open system valve.
  - (d) Loosen pressure gland and insert probe to mark. Be sure probe opening is facing upstream. WEAR HEAVY GLOVES!
  - (e) Tighten pressure gland.
  - (f) Shorten safety chain to secure probe.
7. Slowly open inlet ball valve until fully open.
8. Check visually for leaks - system is pressurized to the outlet regulating valve and correct any leaks.
9. Fill water bucket and insert coils in buckets.

10. Ice the ice bucket and add water to establish coil contact.
11. Open outlet regulating valve slowly and regulate flow to obtain an outlet temperature between 20 and 30°C.

### 13.3.3 Basic Well Sample

1. Flush system with at least three liters of brine while making the following entries on the data sheet:
  - (a) Sample ID, date, start time, air temperature, barometric pressure.
  - (b) Well conditions: pressure, temperature, flow rate.
  - (c) Sample temperature at coil outlet when flow is stabilized.
2. Determine and record pH and exit temperature.
3. Fill, seal, and label RU bottles.
4. Make SiO<sub>2</sub> dilutions by withdrawing 2 ml brine from the RU bottle, add to 100 ml volumetric cylinder and fill to mark with DI water. Empty diluted sample into SiO<sub>2</sub> sample bottle and label.

### 13.3.4 Suspended Solids Collection

1. Close ball valve and insert weighed filter and holder into line - restart flow and collect 5 liters of measured flow using graduated cylinder.
2. From this 5 liter flow, fill FA bottles and FU bottles by directing 1 liter into each bottle. Seal and label bottles.
3. Stop flow, remove and return used filter to its protective container and label container with source, volume of brine, and date. Handle filter carefully.
4. Remove filter holder.

#### Redox Potential

1. If requested, measure redox potential by flowing brine for 5 min, excluding all air!

### 13.3.5 Gas Characterization

1. Collect total CO<sub>2</sub> and H<sub>2</sub>S samples as follows:
  - (a) Attach sparge tube/stopper to coil outlet using Tygon tubing, establish flow.
  - (b) Check temperature.
  - (c) Sparge gas + liquid into CO<sub>2</sub> bottle with NaOH until full to 1 liter mark (be careful to hit mark exactly).
  - (d) Repeat with H<sub>2</sub>S bottle with ZnAc solution.
2. Make H<sub>2</sub>S, NH<sub>3</sub>, and O<sub>2</sub> determinations using field kits. Record data.

#### Gas:Liquid Ratio Procedure

1. Equipment Set-up:
  - (a) Set-up glass separator so that outlet of the separator is 2 to 2-1/2 ft above the ground.
  - (b) Attach Tygon tube from sample cooling coil outlet to the middle connection on the glass separator.
  - (c) Connect top of glass separator to inlet of Wet Test meter with Tygon tubing.
  - (d) Attach Tygon tubing with pinch clamp to bottom connection on the glass separator.
2. With sample water flowing from cooling coils, adjust pinch clamp on bottom of separator to maintain constant water level in separator.
3. Make sure that Wet Test meter dial is working.
4. Zero Wet Test meter by:
  - (a) Setting small dials to zero.
  - (b) Setting large dial to -0.5 liter.
5. When large dial on meter reads "zero", simultaneously:
  - (a) Start stopwatch.

(b) Start collecting brine which should be at a constant temperature of 20 to 25°C.

6. Collect 5 liters of brine from bottom line on separator in the graduated cylinder. Record temperature of brine.
7. When exactly 5 liters of brine has been collected, simultaneously record time (from stopwatch) and gas volume reading from Wet Test Meter.
8. Attach sparger for CO<sub>2</sub> sampling on bottom Tygon tube from glass separator.
9. Measure pH of separated liquid.
10. Collect CO<sub>2</sub> liquid sample in 2 N NaOH for CO<sub>2</sub> analysis of liquid. Label as CO<sub>2</sub> from separated liquid.
11. Disconnect glass separator from sample cooling coils.

#### Total Gas Sample

1. Fill glass bulb (750 ml) with brine (make sure all air bubbles are out). Measure and record brine temperature.
2. Invert bulb and place outlet of bulb into bucket of water.
3. When bulb is full of gas and gas bubbles are seen in the bucket of water, close outlet, then close inlet stopcocks on gas bulb, and disconnect Tygon tube from sample cooling coil. Label bulb.

#### 13.3.6 Shutdown

1. Record pressure, temperature, and flow of well and air temperature at end of sampling.
2. Remove probe and close sample valve on the system.
3. Disconnect flex hose from insertion probe and flush with DI water. Blow the sampling train dry using Tygon tube as mouth piece.
4. Remove pH probe and place in pH 4 buffer.
5. Police area for litter and consolidate sample train.

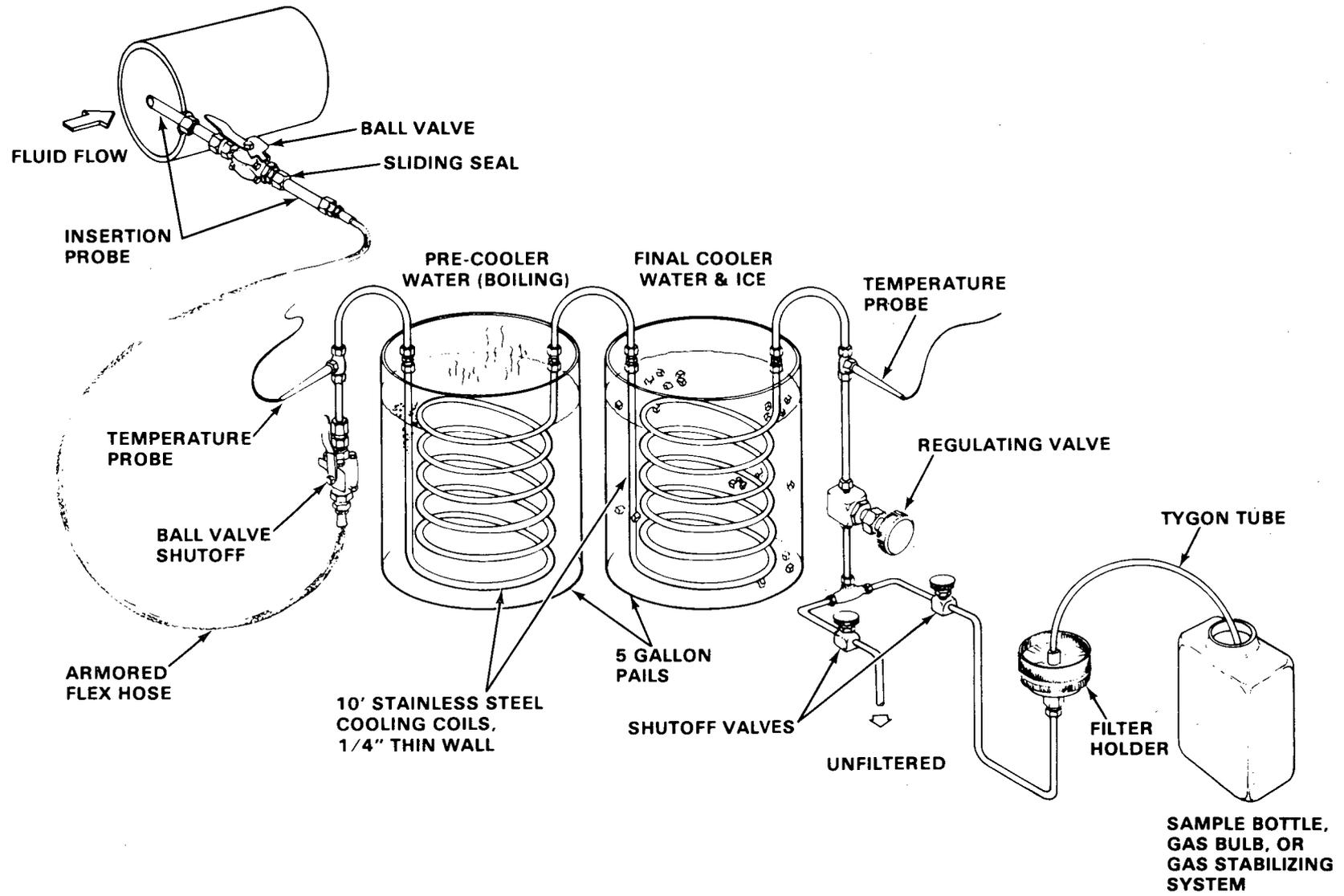


FIGURE 13.1. Geothermal Liquid Sampling Double Coil Assembly

PACIFIC NORTHWEST LABORATORY

SAMPLE DATA

Sample Point I.D. \_\_\_\_\_ Date \_\_\_\_\_

Water (Brine) (at Start) Pressure \_\_\_\_\_ Temp. \_\_\_\_\_ Flow \_\_\_\_\_  
 Well Suppling Brine I.D. Flow

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Air Temperature \_\_\_\_\_

Barometric Pressure \_\_\_\_\_

Start Time \_\_\_\_\_

Coil Outlet Temperature \_\_\_\_\_

pH \_\_\_\_\_ @ \_\_\_\_\_ °C Time \_\_\_\_\_

Conductivity \_\_\_\_\_ @ \_\_\_\_\_ °C Time \_\_\_\_\_

Sample I.D.		Temperature	Time	Comments
_____	RU //	_____	_____	_____
_____	RU //	_____	_____	_____
_____	RU //	_____	_____	_____
_____	SiO <sub>2</sub> //	_____	_____	_____
_____	SiO <sub>2</sub> //	_____	_____	_____
_____	SiO <sub>2</sub> //	_____	_____	_____

Weighed Filter I.D. # \_\_\_\_\_ Time on \_\_\_\_\_ Time off \_\_\_\_\_

Volume through Filter \_\_\_\_\_

_____	FA //	_____	_____	_____
_____	FA //	_____	_____	_____
_____	FA //	_____	_____	_____
_____	FU //	_____	_____	_____
_____	FU //	_____	_____	_____
_____	FU //	_____	_____	_____
_____	CO <sub>2</sub> //	_____	_____	_____
_____	CO <sub>2</sub> //	_____	_____	_____
_____	CO <sub>2</sub> //	_____	_____	_____

_____	H <sub>2</sub> S	//	_____	_____	_____
_____	H <sub>2</sub> S	//	_____	_____	_____
_____	H <sub>2</sub> S	//	_____	_____	_____
_____	Gas	//	_____	_____	_____
_____	Gas	//	_____	_____	_____
_____	Gas	//	_____	_____	_____

Field H<sub>2</sub>S \_\_\_\_\_ Field NH<sub>3</sub> \_\_\_\_\_ Field O<sub>2</sub> \_\_\_\_\_

Redox Potential \_\_\_\_\_ @ Temperature \_\_\_\_\_ Time \_\_\_\_\_

Gas/Liquid Ratio \_\_\_\_\_ @ Temperature \_\_\_\_\_

Time Start \_\_\_\_\_ Litres Collected \_\_\_\_\_ Time Finish \_\_\_\_\_

pH of Separated Liquid \_\_\_\_\_

No. \_\_\_\_\_ CO<sub>2</sub> from Separator (Time) \_\_\_\_\_

Water (Brine)(at end of Sampling) Pressure \_\_\_\_\_ Temperature \_\_\_\_\_

Flow \_\_\_\_\_

Time Completed \_\_\_\_\_ Signed by: \_\_\_\_\_

Remarks:

APPENDIX A

EXAMPLE OF SAMPLE CALCULATION,

FILTER 7-2

APPENDIX A - EXAMPLE OF SAMPLE CALCULATIONS, FILTER 7-2

A.1 FILTRATION

A.1.1 Single Filter

$$\text{Area of Filter } \pi r^2 = (\pi)(4.2 \text{ cm}/2)^2 = 13.85 \text{ cm}^2$$

XRF Concentration of Cl: 17.1  $\mu\text{g}/\text{cm}^2$  Cl Filter 7-2

$$\begin{array}{r} -0.46 \mu\text{g}/\text{cm}^2 \text{Cl Blank Filter} \\ \hline 16.46 \mu\text{g}/\text{cm}^2 \end{array}$$

$$\text{Weight of NaCl: } (16.64 \mu\text{gCl}/\text{cm}^2)(13.85 \text{ cm}^2)(1.65 \text{ NaCl}/\text{Cl}) = 380 \mu\text{gCl}$$

Real Weight Gain Due to Suspended Solids

$$\begin{array}{r} \text{Total Weight Gain: } 0.0017 \text{ g}/5 \ell \\ \text{Minus Salt Weight: } 0.0004 \text{ g}/5 \ell \\ \hline 0.0013 \text{ g}/5 \ell \end{array}$$

$$\text{Total Suspended Solids } (0.0013 \text{ g}/5 \ell)/5 = \underline{\underline{0.26 \text{ mg}/\ell}}$$

A.1.2 Filter Chain

XRF Average Cl for Filters

$$\begin{array}{r} 16.26 \mu\text{g}/\text{cm}^2 \text{Cl Average} \\ 0.46 \mu\text{g}/\text{cm}^2 \text{Cl Blank Filter} \\ \hline 15.80 \mu\text{g}/\text{cm}^2 \text{Cl} \end{array}$$

Weight of NaCl

$$(15.80 \mu\text{gCl}/\text{cm}^2)(13.85 \text{ cm}^2)(1.65 \text{ NaCl}/\text{Cl})(4 \text{ Filters})(10^3 \text{ mg}/\mu\text{g}) = 1.44 \text{ mg}$$

Real Weight Gain Due to Suspended Solids

$$\begin{array}{r} \text{Total Weight of 4 Filters: } 7.2 \text{ mg}/5 \ell \\ \text{Minus Weight of NaCl on 4 Filters: } 1.44 \text{ mg}/5 \ell \\ \hline 5.76 \text{ mg}/5 \ell \end{array}$$

$$\text{Total Suspended Solids } (5.76 \text{ mg}/5 \ell)/5 = \underline{\underline{1.15 \text{ mg}/\ell}}$$

### A.1.3 XRF Data

Total Weight of Elements:	23.81 $\mu\text{g}/\text{cm}^2$
Minus Background - Blank Filter:	<u>1.83 <math>\mu\text{g}/\text{cm}^2</math></u>
Total Weight by XRF	21.98 $\mu\text{g}/\text{cm}^2$

Total Suspended Solid by XRF

$$\begin{aligned} & (21.98 \mu\text{g}/\text{cm}^2)(13.85 \text{ cm}^2)(10^3 \text{ mg}/\mu\text{g}) = 0.304 \mu\text{g Total Weight} \\ & (16.64 \mu\text{gCl}/\text{cm}^2)(13.85 \text{ cm}^2)(10^3 \text{ mg}/\mu\text{g}) \\ & = 0.230 \text{ mg Weight Cl} \\ & = 0.074 \text{ mg Weight Suspended Solids} \end{aligned}$$

Total Suspended Solid 0.074 mg for 5 $\ell$  = 0.015 mg/ $\ell$

### A.1.4 Laser Data

$$\begin{aligned} \text{Total Mass/cc} &= (\text{Volume of Particle})(\text{Density})(\text{No. of Particles/cc}) \\ &= (4/3 \pi r^3)(3 \text{ g/cc}) N \\ &= (4/3 \pi \frac{d^3}{3})(3 \text{ g/cc}) N \\ &= (1/6 \pi d^3)(3 \text{ g/cc}) N \text{ for } d \text{ in } \mu\text{m, than} \\ &= (\pi/6)(d \times 10^{-4} \text{ m})^3(3 \text{ g/cc}) N \\ &= 1.57 \times 10^{-12} d^3 N \end{aligned}$$

where N = No. of Particles/cc

$$d^3 = (\text{Average Diameter})^3$$

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