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# Collection of geothermal fluids for chemical analysis

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# 1 Introduction

The collection of samples for chemical analysis is the first step in a long process which eventually yields results that provide building blocks in the model of a geothermal system. It is imperative that this step is properly carried out because all subsequent steps depend on it.

There are several hidden dangers inherent in the collection of geothermal sample. The terrain may be treacherous and dangerous chemicals need to be handled.

Thus there is an obvious need for well trained personnel with insight into possible errors and interferences to carry out this task.

The most common mistakes made during sampling involve the use of improper containers, improper cleaning and lack of or improper treatment for the preservation of samples.

This report on the collection of geothermal fluids is an update of a previous one describing methods used at Orkustofnun and now at Iceland GeoSurvey (ISOR) (Ólafsson, 1988). Some methods described by Sheppard and Giggenbach (1985) have also been considered.

Recently Arnórsson et al. (2006) have described similar methods for sampling and analysis of geothermal fluids as the ones outlined here.

## 2 Containers

For lightness, ruggedness and tolerance of bumps in the field plastic bottles are the best. Most plastics are however relatively permeable and let atmospheric air easily through, possibly setting off oxidation reactions, and liquids may easily evaporate through them causing concentration of constituents and possible oversaturation. Many plastics are also rife with possible adsorption sites for sample constituents and may thus decrease their concentrations.

Glass is fragile and relatively heavy but can relatively easily be made airtight. Thus glass containers are preferable for the preservation of constituents affected by atmospheric air. Constituents that are sensitive to light are collected into amber bottles.

If containers have not been specifically pre-cleaned and prepared for a certain task they should be rinsed at least three times with the sample fluid prior to collection.

## 3 Preservation of samples

Some constituents will not survive intact from sample collection to analysis without special precautions. Common reasons for concentration changes are interaction with suspended matter, adsorption on the walls of the containers, biological activity, redox reactions, polymerization and precipitation. Different preservation methods are needed for the various processes and thus the total sample will comprise several sub-

samples. Preservation methods may be physical or chemical and the more common ones are listed in Table 1.

It is desirable that samples be kept relatively cool apart from the inconvenience of handling boiling hot water and steam. Fluid that is well above ambient temperature is therefore cooled to ambient temperature using a cooling device, usually a cooling coil immersed in cold water, during collection. Steam samples collected into NaOH in double-port bottles may by-pass the cooling device and the bottle itself be cooled in cold water.

**Table 1.** *Preservation methods for geothermal samples.*

| Type     | Method             | Purpose  | Used for   |
|----------|--------------------|--|--|
| Physical | Filtration         | Prevent interaction with suspended matter  | Anions, cations  |
|          | Freezing           | Prevent biological activity  | Nutrients  |
|          | Airtight container | Prevent interaction with atmospheric air   | Volatiles  |
|          | On-site analysis   | Prevent reactions of reactive constituents   | Reactive constituents  |
| Chemical | Base addition      | Absorption of acid gases   | CO <sub>2</sub> , H <sub>2</sub> S in steam, δ <sup>34</sup> S in H <sub>2</sub> S in vapour           |
|          | Acidification      | Prevent adsorption on walls of containers  | Cations  |
|          | Precipitation      | Prevent a constituent from reaction to change the concentration of another             | Sulphide to preserve sulphate  |
|          | Sterilization      | Prevent biological activity, using HgCl or formaldehyde                                | δ <sup>34</sup> S and δ <sup>18</sup> O in SO <sub>4</sub> , prevents biological oxidation of sulphide |
|          | Dilution           | Prevent polymerization and precipitation   | Silica   |
|          | Redox              | To change oxidation state of a volatile constituent to make it less volatile           | Hg   |
|          | Ion exchange       | Concentrate and further prevent adsorption on walls of container of trace constituents | Trace cations  |
|          | Extraction         | Concentrate and further prevent adsorption on walls of container of trace constituents | Trace cations  |

## 4 Types of samples

Geothermal samples may consist of water, gas, a mixture of water and steam, and steam only (water vapour + non-condensable gases). The sample sources may be geothermal wells, hot springs or pools, and fumaroles.

## 4.1 Collection of samples from hot springs and hot-water wells

The collection of samples of non-boiling water can be divided into two categories, samples from natural springs and samples from hot water wells. When collecting samples from hot springs it is desirable that the water be free-flowing from the sample spot. If not, a sampling pump is needed. Record water temperature and discharge as well as wellhead pressure if available.

An untreated sample is collected for pH, carbonate; H<sub>2</sub>S and conductivity measurements as well as for the analysis of Mg and SiO<sub>2</sub>. If the SiO<sub>2</sub> concentration is expected to be more than 100 ppm, the sample is diluted with distilled and deionized water to an ideal concentration of 30 to 100 ppm in triplicate and the dilution factor recorded. A filtered sample is collected for anions and a filtered and acidified sample is collected for cations. In cases of high sulphide concentrations the sulphide is precipitated with Zn(CH<sub>3</sub>COO)<sub>2</sub> and SO<sub>4</sub>. analysed for from a filtered sample. For the analyses of stable isotopes ( $\delta^2\text{H}$ ,  $\delta^{18}\text{O}$ ) the isotope laboratory needs 60 ml of filtered sample. The various sub-samples collected are described in detail in Table 2.

**Table 2.** Treatment and sub-samples from hot springs and hot-water wells.

| Treatment  | Container                  | Specification | To determine   |
|--|----------------------------|---------------|--|
| None; amber glass bottle with ground glass stopper   | 250–300 ml<br>Glass        | Ru            | pH, CO <sub>2</sub> , H <sub>2</sub> S (if not in field), conductivity               |
| None   | 200 ml<br>plastic          | Ru            | Mg, SiO <sub>2</sub> if < 100 ppm  |
| Dilution; 50 ml of sample added to 50 ml of distilled, deionized water   | 3x100 ml<br>plastic        | Rd (1:1)      | SiO <sub>2</sub> if > 100 ppm  |
| Filtration   | 200 ml<br>plastic          | Fu            | Anions   |
| Filtration; 0.8 ml conc. HNO <sub>3</sub> (Suprapur) added to 200 ml sample  | 200 ml<br>plastic          | Fa            | Cations  |
| Filtration; 2 ml 0.2 M ZnAc <sub>2</sub> added to sample in 100 ml volumetric glass flask and $\geq 10$ ml to $\geq 500$ ml bottle containing $\geq 25$ mg SO <sub>4</sub> to precipitate sulphide | 100 ml, >500 ml<br>plastic | Fp, Fpi       | SO <sub>4</sub> , $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$ in SO <sub>4</sub> |
| Filtration; 1 60 ml and 2 1000 ml amber glass bottles  | 60 ml, 1000 ml<br>Glass    | Fui, Fuc, Fut | $\delta^2\text{H}$ , $\delta^{18}\text{O}$ , $^{13}\text{C}$ , $^3\text{H}$          |

## 4.2 Collection of samples from high-temperature wells and fumaroles

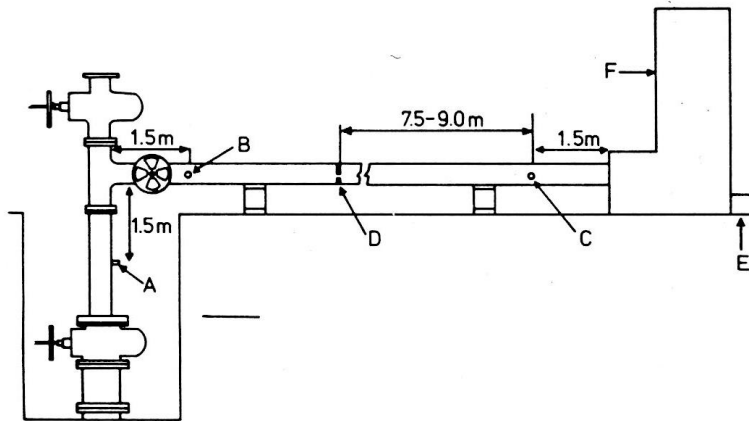
The collection of representative samples from high-temperature drillholes is a complex procedure. It is done either by using the separator on the wellhead separating the whole discharge or with a small Webre separator. Natural steam discharge may occur in many different forms, such as gentle discharge from a large area of hot ground or major discharge from large fumaroles. The most useful information is often obtained from steam discharged from powerful fumaroles.

### 4.2.1 High-temperature wells

**Table 3.** *Treatment and sub-samples from two-phase geothermal wells.*

| Phase  | Treatment  | Specification  | To determine   |
|--------|--|----------------|--|
| Vapour | None; amber glass bottle   | Ru             | $\delta^2\text{H}$ , $\delta^{18}\text{O}$   |
|        | 0.5 ml 0.2 M $\text{ZnAc}_2$ added to sample in 100 ml volumetric glass flask to precipitate sulphide  | Rp             | $\text{SO}_4$  |
|        | None   | Ru             | Anions   |
|        | 0.8 ml conc. $\text{HNO}_3$ (Suprapur) added to 200 ml sample  | Ra             | Cations  |
|        | Added to 50 ml 40% $\text{NaOH}$ in evacuated doubleport bottle  | Gas sample, Ai | $\text{CO}_2$ , $\text{H}_2\text{S}$ in $\text{NaOH}$ , residual gases in gas phase, $\delta^{34}\text{S}$ in $\text{H}_2\text{S}$ in vapour |
| Liquid | None   | Ru             | $\text{Mg}$ , $\text{SiO}_2$ if < 100 ppm  |
|        | Dilution; 10 ml of sample added to 90 ml of distilled, deionized water   | Rd (1:10)      | $\text{SiO}_2$ if > 100 ppm  |
|        | None; amber glass bottle with ground glass stopper   | Ru             | pH, $\text{CO}_2$ , $\text{H}_2\text{S}$ (if not in field)   |
|        | Filtration   | Fu             | Anions   |
|        | Filtration; 2 ml 0.2 M $\text{ZnAc}_2$ added to sample in 100 ml volumetric glass flask and $\geq 10$ ml to $\geq 500$ ml bottle containing $\geq 25$ mg $\text{SO}_4$ to precipitate sulphide | Fp, Fpi        | $\text{SO}_4$ , $\delta^{34}\text{S}$ and $\delta^{18}\text{O}$ in $\text{SO}_4$   |
|        | Filtration; 1 60 ml and 2 1000 ml amber glass bottle, with ground glass stoppers   | Fui, Fuc, Fut  | $\delta^2\text{H}$ , $\delta^{18}\text{O}$ , $^{13}\text{C}$ , $^3\text{H}$  |
|        | Filtration; 0.8 ml conc. $\text{HNO}_3$ (Suprapur) added to 200 ml sample  | Fa             | Cations  |

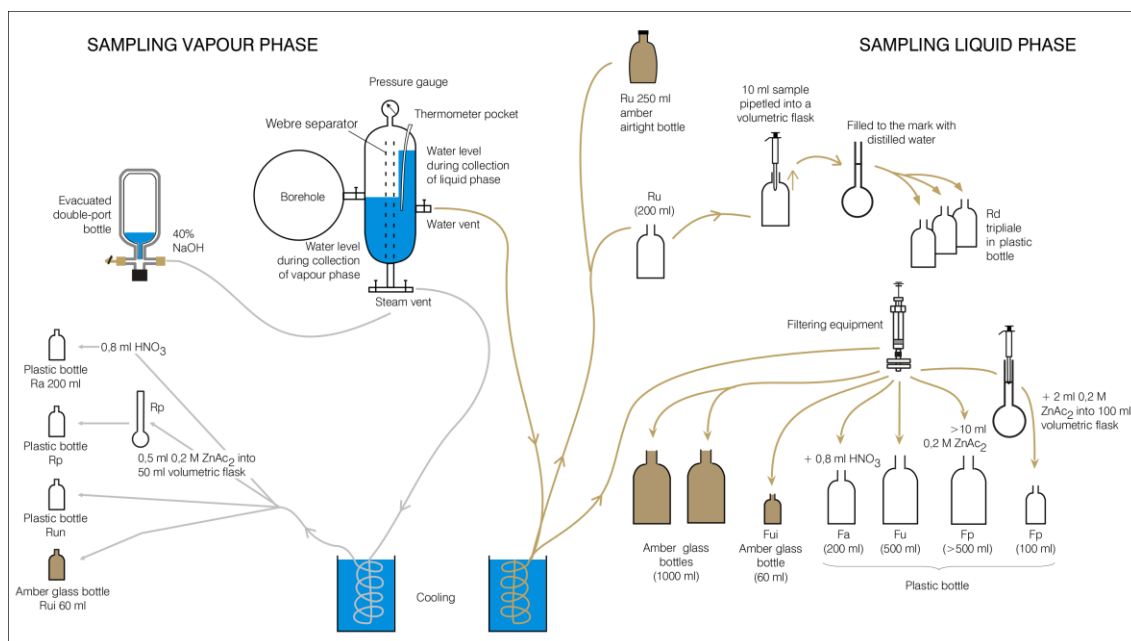




**Figure 1.** The positions of sampling points on the surface piping of a geothermal well from which representative steam and water samples are collected. A, B and C, sample points; D, constriction such as back pressure plate or valve; F, silencer; E, weir box of silencer (Ellis and Mahon 1977).

|   |   |
|---|---|
|   |   |
| <p>Webre separator at wellhead</p>              | <p>Webre separator adjusted at wellhead</p>     |
|   |   |
| <p>Webre separator ready for water sampling</p> | <p>Webre separator ready for steam sampling</p> |

**Figure 2.** Webre separator connected and adjusted at wellhead.



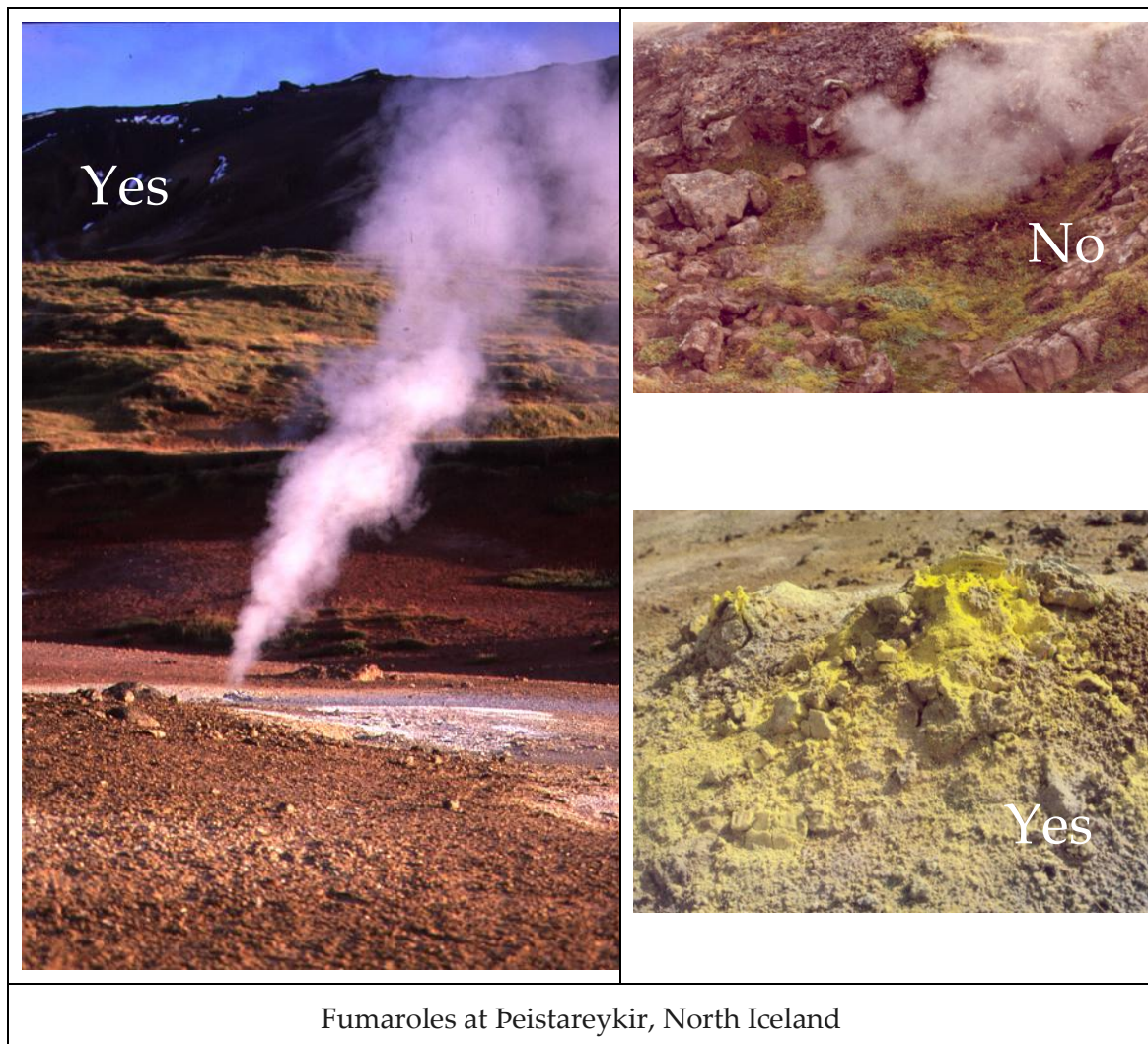
**Figure 3.** Overview of collection of a sample from a two-phase geothermal well for chemical analysis.

It has been shown that the most representative samples are collected from the flow of a two-phase well at about 1.5 m distance from the T-joint at the well top (Figures 1 and 2). The various sub-samples collected are described in detail in Table 2 but the total procedure is shown in Figure 3. Samples are collected into plastic bottles unless otherwise specified.

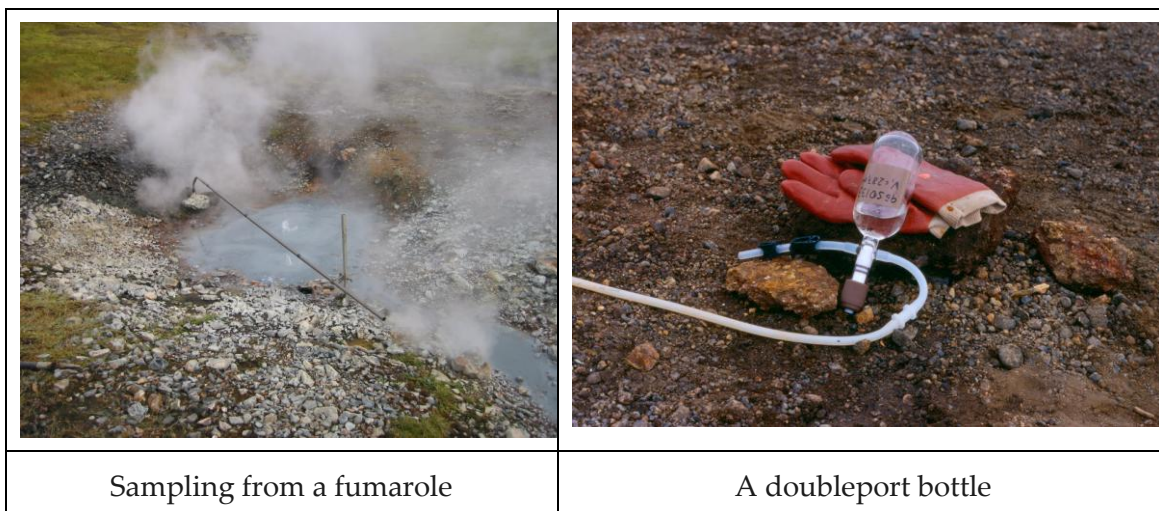
The vents on the Webre separator are opened and the fluid is allowed to flow from the borehole through the separator. Care is taken that the pressure in the separator does not deviate much from that of the wellhead. For the collection of the vapour phase the water level inside the separator is kept low until preferably a mixture of water and steam issues through the water vent. A blue cone should form at the steam vent showing that dry steam is being issued. To check the efficiency of the separation a small sample of condensed steam may be drawn and the concentration of a non-volatile component such as Na or Cl determined, compared with the concentration of the same component in the liquid phase and the percentage of carry-over calculated. A sample from a one-phase steam well is drawn in the same way as the vapour fraction of the sample from the two-phase well except that of course no separator is necessary. A sample from a one phase water well is similarly drawn without a separator and is analogous to the liquid phase sample from the two-phase well. If  $t < 70^\circ\text{C}$  it may be desirable to determine the dissolved oxygen concentration of the water to estimate its corrosion potential. This determination is carried out during sampling as described below.

#### 4.2.2 Fumaroles and springs

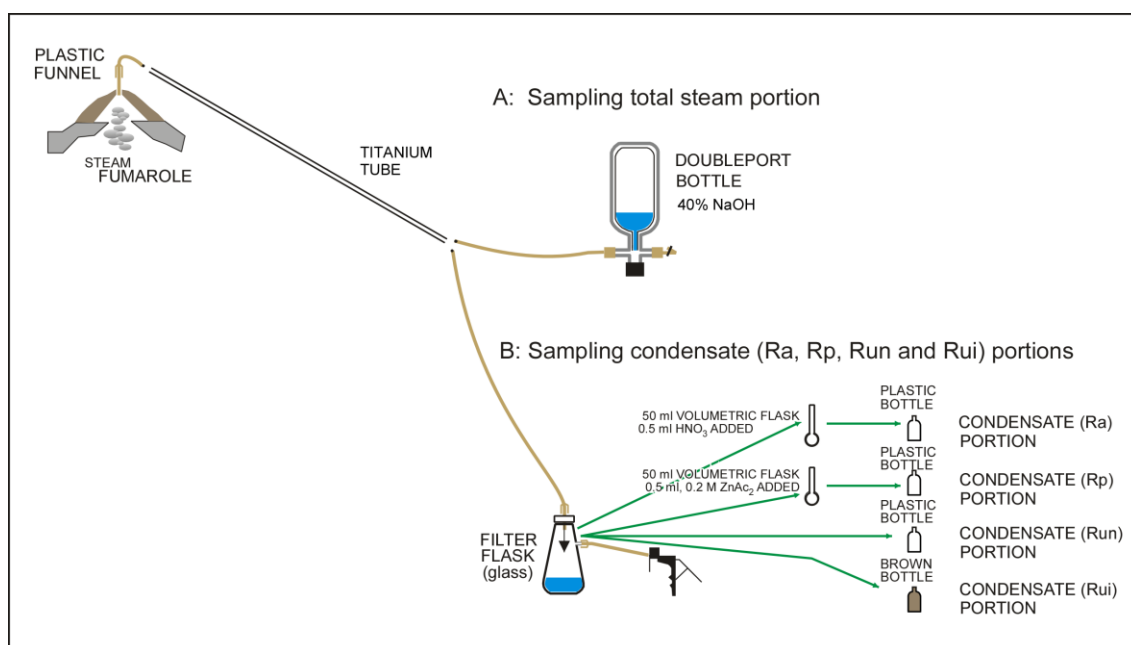
*Fumaroles:* Care has to be taken that a discrete, directed outflow is chosen and diffuse ones avoided at all costs (Figure 4). A good guide to the suitability of an outflow for sampling is sulphur deposits. A funnel is placed atop the outflow and care taken that no atmospheric air is drawn in. The funnel is connected to a titanium tube which is directed to a lower point where the sample is collected (Figures 5 and 6). The sub-samples are the same as those collected of the vapour phase of a two-phase well sample.



**Figure 4.** *Suitability of fumaroles for sample collection.*



**Figure 5.** *Sampling a fumarole.*



**Figure 6.** *Collection of steam sample from a fumarole.*

*Springs.* Care has to be taken to obtain a sample as near to the outflow as possible. An indicator such as ink may be used if it is difficult to find. Normally the water sample will be drawn with a pump into an evacuation flask. The filtering apparatus is fitted between the sample and the pump when appropriate. If a gas sample is required two evacuated flask one with taps on both ends below and a doubleport gas bottle containing 40% NaOH above are arranged in series. The taps are opened slowly, first on the two-ended flask and care taken that water does not enter the doubleport bottle (Figure 7).

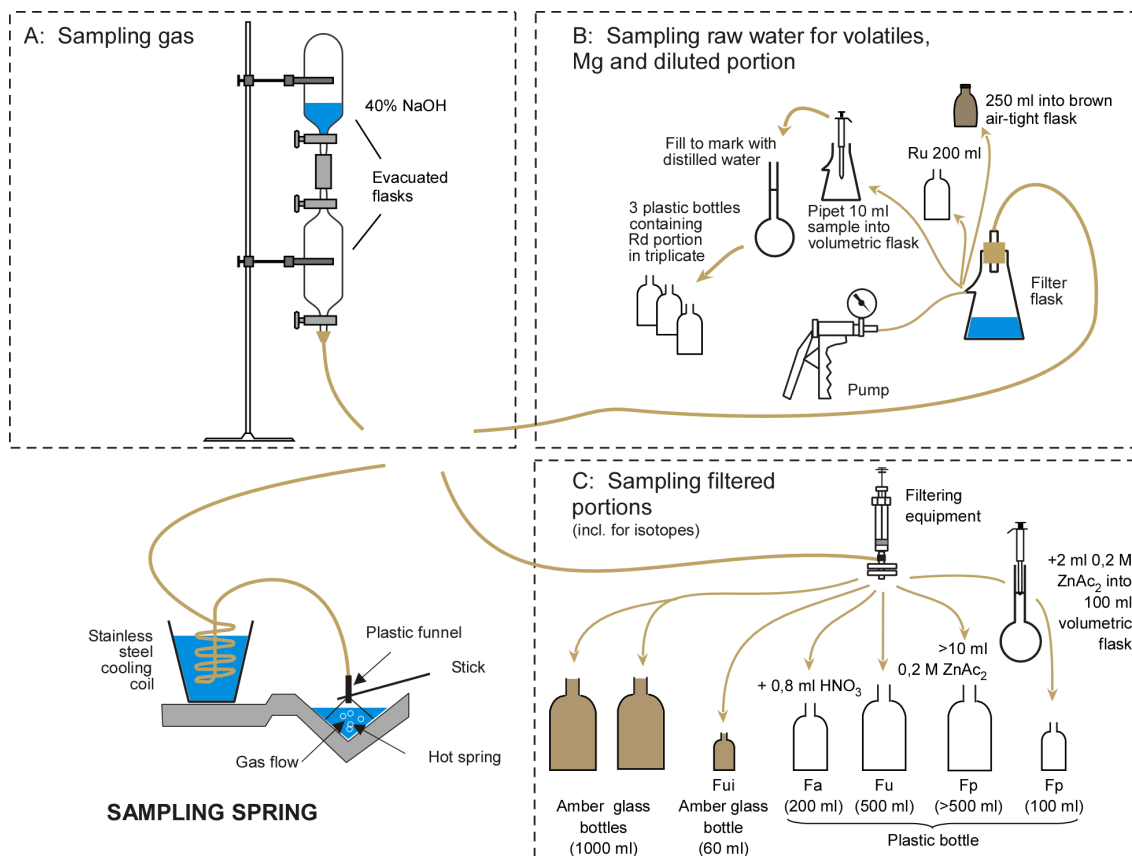
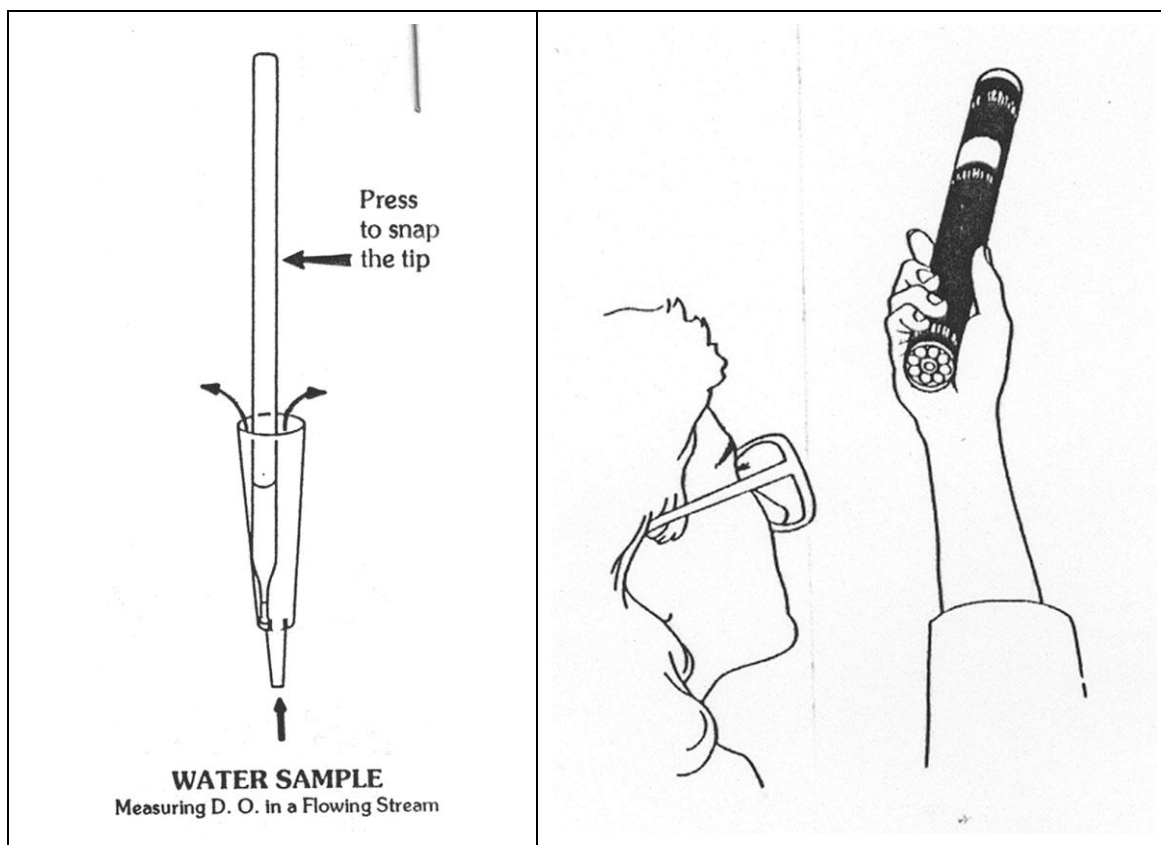


Figure 7. Collection of sample from a spring.

## 5 On site analysis

Some constituents are extremely reactive and if possible it is advisable to analyse for them on-site. As mentioned above the determination of dissolved oxygen in water from relatively cool wells should be carried out during sampling. It is also desirable to carry out analysis for hydrogen sulphide in the field. Initial characterization of the fluid may be important so pH and conductivity are regularly determined in the field and a simple colorimeter is sometimes in the luggage for immediate silica analysis. Here it is recommended that analysis for oxygen and hydrogen sulphide be carried out in the field.

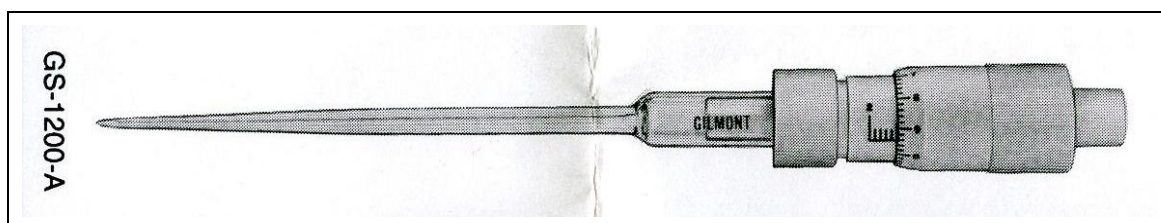
*Oxygen determination.* Use ampoules from CHEMetrics, Inc., containing Rhodazine D for concentrations 0-100 ppb, but Indigo carmine for higher concentrations. Purge the flow of any air bubbles by using the fastest rate possible for the water. Break the tip of the ampoule and fill it with water. Mix well and compare to pre-prepared colours in a comparator and read the O<sub>2</sub>-concentration (Figure 8).



**Figure 8.** *The determination of dissolved oxygen*

*Hydrogen sulphide determination.* Reagents: 5N NaOH, acetone, solid dithizone, 0.001 M mercury acetate ((CH<sub>3</sub>COO)<sub>2</sub>Hg). Add 5 ml NaOH solution to a 50 ml volumetric flask and fill to the mark with sample. Place 0.1-5 ml in an Erlenmeyer flask, add 5 ml acetone and bring the volume to 10 ml with distilled, deionized water. Add a spec of dithizone with a spatula. Titrate with 0.001 M HgAc<sub>2</sub> using a 2 ml microburette (Figure 9), graduated to 0.001 ml intervals, to a pink end point. Record the titre and calculate H<sub>2</sub>S concentration according to the following equation:

$$\text{H}_2\text{S (mg/L)} = (\text{ml HgAc}_2 * 34) / \text{ml sample.}$$



**Figure 9.** *Microburette for the titration of H<sub>2</sub>S*

## 6 Checklist and recording of information

A checklist showing materials and equipment that needs to be taken along for the collection of a sample from a high-temperature geothermal well is depicted in Table 3. All information regarding each sample obtained during sampling and analysis should be recorded on a recording sheet such as the one in Figure 10.

**Table 3.** *Checklist for sampling high-temperature geothermal well*

- ⊙ Webre separator
- ⊙ Thermometer for separator
- ⊙ Pressure gauges
- ⊙ Sampling hoses
- ⊙ Cooling device
- ⊙ Tools, Fittings; Teflon tape
- ⊙ Evacuated double-port bottle
- ⊙ Plastic bottles
- ⊙ Glass bottles
- ⊙ Filtering apparatus
- ⊙  $\text{ZnAc}_2$  and  $\text{HNO}_3$
- ⊙ Pipettes
- ⊙ Volumetric flasks
- ⊙ Distilled water
- ⊙ Field record card and permanent marker
- ⊙ Sample number
- ⊙ Vinyl gloves
- ⊙ Overalls
- ⊙ Ear mufflers
- ⊙ Face guard

### A. Vapour phase

- A.1. Condensate for Na, Cl or B in 100 ml plastic bottle
- A.2. Condensate for stable isotopes 60 ml amber glass bottle
- A.3. Steam in an evacuated doubleport bottle
- (A.4. Condensate for total analysis)

### B. Water phase

- B.1. Ru in 250 ml amber glass bottle
- B.2. Rd (1:10) sample for  $\text{SiO}_2$  in three 100 ml plastic bottles
- B.3. Fu 200 ml plastic bottle
- B.4. Fa (0.8 ml  $\text{HNO}_3$ ) in 200 ml plastic bottle
- B.5. Fp (2ml 0.2M  $\text{ZnAc}_2$ ) in 100 ml plastic bottle, Fpi in  $\geq 500$  ml plastic bottle

- B.6. Fui for stable isotopes of oxygen and hydrogen ( $^{18}\text{O}$ ,  $^2\text{H}$ ) in 60 ml amber glass bottle
- B.7. Fuc for  $^{13}\text{C}$
- B.8. Fut for tritium ( $^3\text{H}$ )

**Record pressure and temperature of separator at sampling**

**ISOR**

Sample No. \_\_\_\_\_  
 Date/Time \_\_\_\_\_  
 Site/Site classification \_\_\_\_\_  
 Location \_\_\_\_\_  
 Sampled by \_\_\_\_\_

Project No./Supervisor \_\_\_\_\_  
 Coordinates \_\_\_\_\_  
 Site Description \_\_\_\_\_

**RECORDS DURING SAMPLING**

Depth (m) \_\_\_\_\_  
 Temperature °C \_\_\_\_\_  
 Flow/Estimated flow (kg/s) \_\_\_\_\_  
 Pressure (Psi) \_\_\_\_\_  
 Conductivity ( $\mu\text{S}/\text{cm}/^\circ\text{C}$ ) \_\_\_\_\_  
 Wellhead Pressure (Po) \_\_\_\_\_  
 Enthalpy (kJ/kg) \_\_\_\_\_  
 Discharge (kg/sec) \_\_\_\_\_

pH/°C \_\_\_\_\_  
 O<sub>2</sub> \_\_\_\_\_  
 H<sub>2</sub>S \_\_\_\_\_

Notes \_\_\_\_\_

**SUBSAMPLES (NO.)      SAMPLE HANDLING**

|                |       |               |                          |                          |
|----------------|-------|---------------|--------------------------|--------------------------|
| Ru             | _____ | Filters       | 0,45                     | 0,20                     |
| Rd             | _____ | not known     | <input type="checkbox"/> | <input type="checkbox"/> |
| Ra             | _____ | steel/acetate | <input type="checkbox"/> | <input type="checkbox"/> |
| Fu             | _____ | PTFE/acetate  | <input type="checkbox"/> | <input type="checkbox"/> |
| Fa             | _____ | PTFE/nitrate  | <input type="checkbox"/> | <input type="checkbox"/> |
| Fp             | _____ | PTFE/nitrate  | <input type="checkbox"/> | <input type="checkbox"/> |
| Trace elements | _____ | glass         | <input type="checkbox"/> | <input type="checkbox"/> |
| Isotopes       | _____ | plastic       | <input type="checkbox"/> | <input type="checkbox"/> |

**CHEMICAL COMPOSITION OF LIQUID FRACTION (mg/kg)**

pH/°C \_\_\_\_\_  
 CO<sub>2</sub> \_\_\_\_\_  
 H<sub>2</sub>S \_\_\_\_\_  
 NH<sub>3</sub> \_\_\_\_\_  
 B \_\_\_\_\_  
 Cond ( $\mu\text{S}/\text{cm}/^\circ\text{C}$ ) \_\_\_\_\_  
 SiO<sub>2</sub> \_\_\_\_\_  
 TDS \_\_\_\_\_  
  $\delta\text{D}(\text{‰})$  \_\_\_\_\_  
  $\delta\text{O}^{18}(\text{‰})$  \_\_\_\_\_  
 Na \_\_\_\_\_  
 K \_\_\_\_\_  
 Mg \_\_\_\_\_  
 Ca \_\_\_\_\_  
 Sr \_\_\_\_\_  
 F \_\_\_\_\_  
 Cl \_\_\_\_\_  
 Br \_\_\_\_\_  
 NO<sub>3</sub> \_\_\_\_\_  
 SO<sub>4</sub> \_\_\_\_\_  
 Al \_\_\_\_\_  
 Cr \_\_\_\_\_  
 Mn \_\_\_\_\_  
 Fe \_\_\_\_\_  
 Cu \_\_\_\_\_  
 Zn \_\_\_\_\_  
 As \_\_\_\_\_  
 Cd \_\_\_\_\_  
 Hg \_\_\_\_\_  
 Pb \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**CHEMICAL COMPOSITION OF NON-CONDENSABLE GAS (% volume)**

H<sub>2</sub> \_\_\_\_\_  
 Ar \_\_\_\_\_  
 O<sub>2</sub> \_\_\_\_\_  
 N<sub>2</sub> \_\_\_\_\_  
 CH<sub>4</sub> \_\_\_\_\_  
 Rn (dpm/l) \_\_\_\_\_  
 lg/kgcond. \_\_\_\_\_  
 Temperature(°C) \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**CHEMICAL COMPOSITION OF VAPOUR (mg/kg)**

CO<sub>2</sub> \_\_\_\_\_  
 H<sub>2</sub>S \_\_\_\_\_  
 Rn (dpm/kg) \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

**CHEMICAL COMPOSITION OF CONDENSATE (mg/kg)**

pH/°C \_\_\_\_\_  
 CO<sub>2</sub> \_\_\_\_\_  
 H<sub>2</sub>S \_\_\_\_\_  
 NH<sub>3</sub> \_\_\_\_\_  
 B \_\_\_\_\_  
 Na \_\_\_\_\_  
 Cl \_\_\_\_\_  
  $\delta\text{D}(\text{‰})$  \_\_\_\_\_  
  $\delta\text{O}^{18}(\text{‰})$  \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

|        |    |     |     |     |     |     |     |     |     |     |     |  |  |  |  |
|--------|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|--|--|--|--|
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**Figure 10.** Recording sheet for geothermal sample

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