

Designation: E 1675 - 95a

# Standard Practice for Sampling Two-Phase Geothermal Fluid for Purposes of Chemical Analysis<sup>1</sup>

This standard is issued under the fixed designation E 1675; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

- 1.1 The purpose of this practice is to obtain representative samples of liquid and steam as they exist in a pipeline transporting two-phase geothermal fluids.
- 1.1.1 The liquid and steam samples are collected and properly preserved for subsequent chemical analysis in the field or an off-site analytical laboratory.
- 1.1.2 The chemical composition data generated from the analysis of liquid and steam samples may be used for many applications important to geothermal energy exploration, development, and the long-term managed exploitation of geothermal resources. These applications include, but are not limited to, resource evaluations such as determining reservoir temperature and the origin of reservoir fluids, compatibility of produced fluids with production, power generation and reinjection hardware exposed to the fluids (corrosivity and scale deposition potential), long-term reservoir monitoring during field exploitation, and environmental impact evaluations including emissions testing.
- 1.1.2.1 To fully utilize the chemical composition data in the applications stated in 1.1.2, specific physical data related to the two-phase discharge, wellbore, and geothermal reservoir may be required. Mathematical reconstruction of the fluid chemistry (liquid and steam) to reservoir conditions is a primary requirement in many applications. At a minimum, this requires precise knowledge of the total fluid enthalpy and pressure or temperature at the sample point. Fluid reconstruction and computations to conditions different from the sample collection point are beyond the scope of this practice.
- 1.2 This practice is limited to the collection of samples from two-phase flow streams at pressures greater than 10 psig and having a volumetric vapor fraction of at least 20 %. This practice is not applicable to single-phase flow streams such as pumped liquid discharges at pressures above the flash point or

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E-44 on Solar, Geothermal, and Other Alternative Energy Sources and is the direct responsibility of

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Subcommittee E44.15 on Geothermal Field Development.

superheated steam flows. Refer to Specification E 947 for sampling single-phase geothermal fluids.

- 1.3 The sampling of geothermal fluid two-phase flow streams (liquid and steam) requires specialized sampling equipment and proper orientation of sample ports with respect to the two-phase flow line. This practice is applicable to wells not equipped with individual production separators.
- 1.4 In many cases, these techniques are the only possible way to obtain representative steam and liquid samples from individual producing geothermal wells. The sampling problems that exist include the following:
- 1.4.1 Unstable production flow rates that have a large degree of surging,
- 1.4.2 Unknown percentage of total flow that is flashed to steam or is continuously flashing through the production system,
- 1.4.3 Mineral deposition during and after flashing of the produced fluid in wellbores, production piping, and sampling trains,
- 1.4.4 Stratification of flow inside the pipeline and unusual flow regimes at the sampling ports, and
  - 1.4.5 Insufficient flash fraction to obtain a steam sample.
- 1.5 This practice covers the sample locations, specialized sampling equipment, and procedures needed to obtain representative liquid and steam samples for chemical analysis.
- 1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Section 7.

## 2. Referenced Documents

2.1 ASTM Standards:

E 947 Specification for Sampling Single-Phase Geothermal Liquid or Steam for Purposes of Chemical Analysis<sup>2</sup>

2.2 Other Document:

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 12.02.



ASME Code Section VIII, Division 1(1986), Pressure Vessel Design, Fabrication and Certification<sup>3</sup>

## 3. Summary of Practice

3.1 Samples are collected from a pipeline carrying twophase geothermal fluids by using a sampling separator that separates liquid and steam phases through centrifugal force. A fraction of the separated steam is condensed and a fraction of the separated liquid is cooled. Portions of the condensed steam and cooled liquid are collected in appropriate sample containers for subsequent chemical analysis.

# 4. Significance and Use

- 4.1 The objective of this practice is to obtain representative samples of the steam and liquid phases as they exist in the pipeline at the sample point, without allowing steam condensation or additional liquid flashing in the separator. A significant feature of the practice is the use of a cyclone-type separator for high-efficiency phase separation which is operated at flow rates high enough to prevent significant heat loss while maintaining an internal pressure essentially the same as the pipeline pressure.
- 4.2 Another significant feature of the practice is to locate the sampling separator at a point on the pipeline where the two-phase flow is at least partially stratified to aid in the separation process. It is neither necessary nor possible to pass representative proportions of each phase through the sampling separator to obtain representative samples. The separator is usually attached to an appropriately oriented port to collect each specific phase—normally on top of the line for steam and at the bottom for liquid. In some cases, piping configurations can generate unusual flow regimes where the reverse is required. If the ratio of one phase to another is not extreme, representative samples of each phase can often be obtained from a horizontal port on the side of the pipeline.
- 4.3 This practice is used whenever liquid or steam samples, or both, must be collected from a two-phase discharge for chemical analysis. This typically includes initial well-testing operations when a well is discharged to the atmosphere or routine well production when a well discharges to a fluid gathering system and power plant. The combined two-phase flow of several wells producing through a common gathering system may also be sampled in accordance with this practice.
- 4.4 This practice is not typically employed when individual wells produce to dedicated production separators. In these cases, the separated steam and liquid at the outlet of the production separator is sampled in accordance with single-phase sampling methods (Specification E 947).

## 5. Sample Location

5.1 Sample locations vary and are dependent upon the gross quantities of each phase at the sample point. If sample ports are properly oriented on the two-phase pipeline, a certain degree of phase stratification will have occurred prior to sampling, facilitating further separation of the target phase through the sampling separator.

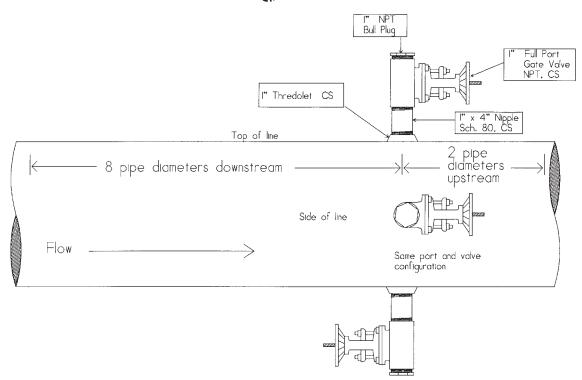
- 5.2 Ports are ideally located on the top and bottom of the pipeline at least eight diameters downstream and two diameters upstream of major flow disturbances such as pipe bends, reductions, valving, etc. (see Fig. 1).
- 5.2.1 In cases where the fluid contains substantial quantities of solid debris that may plug the sample port, the liquid port can be located at a  $45^{\circ}$  angle from the bottom, provided that a sufficient liquid phase is present.
- 5.2.2 If the flow regime is known, the number of ports may possibly be reduced to a single port located either on the side, top, or bottom of the two-phase pipeline. Sufficient quantities of each phase must be available at the single port to allow collection of representative steam and liquid samples.
- 5.2.3 The sample ports must be at least 1-in. diameter and configured with a full-open port ball or gate valve. This requirement is necessary to ensure that only a minimal pressure drop occurs through the port valve and associated piping. Scale and debris often reduce the effective inner diameter of the port, therefore smaller ports are not recommended. The port size restriction also provides a safety margin given the weight of the separator and force needed to install and remove fittings from the port.
- 5.3 Sample ports should never be located on side-stream piping from the main flow line unless only the side-stream fluids are to be characterized. The proportions of each phase are not likely to remain the same in a flow stream split off from the main flow line. Any pressure reduction in the side stream piping will change the steam and liquid compositions to an unknown degree.

#### 6. Equipment

- 6.1 Sampling Separator—A cyclone-type separator rated to the pipeline pressure at the sample point, including a pressure gage, temperature probe, and sight glass (optional). The separator should be designed to attach directly to the sample port to minimize heat loss and pressure drop.
- 6.1.1 A typical sampling separator is shown in Fig. 2. This is a cyclone-type separator with a 1-in. pipe inlet attached at a tangent to the separator body. The separator is rated to 500 psig at 500°F. A pressure gage and thermocouple are located at the top of the separator, and steam and liquid sample valves are located at the bottom. Steam is drawn from the top of the separator through an axial pipe extending up from the bottom of the vessel. Liquid is drawn directly off the bottom. Internal baffles prevent liquid films from rising up the inner walls of the vessel with the steam flow to the sample valves. Vortex breakers are placed in the bottom of the vessel to prevent steam entrainment in the liquid flow to the sample valves.
- 6.1.1.1 The vent valve on the side of the sampling separator (No. 2 in Fig. 2) can be used to maintain an excess flow of steam and liquid through the separator, beyond the amount needed for sample collection. If sufficient quantities of each phase are present, the side vent valve will maintain a liquid level about 2 in. above the liquid sample valve (No. 5 in Fig. 2). This allows collection of both steam and liquid samples from the separator without the need to adjust the liquid level.
- 6.1.1.2 An optional sight-glass (PFA-fluorocarbon) for liquid level is located along one side of the separator to aid in proper separator operation and confirm the position of the

<sup>&</sup>lt;sup>3</sup> Available from American Society of Mechanical Engineers 345 E. 47th St. New York, NY 10017.





Note 1—Minimum pipe diameters required upstream and downstream of major flow disturbances (piping bends, reductions).

FIG. 1 Two-Phase Flowline Sampling Separator Ports

liquid level. The sight glass is only rated to 250 psig and must be removed for higher pressure operation.

6.2 Sample Hoses—Sample hoses are PFA-lined stainless steel braided hoses rated to 500 psig and 450°F. JIC type fittings or quick-disconnect fittings attach hoses to the separator and condenser. Hoses are dedicated to either steam or liquid service to prevent cross-contamination. The inner diameter of the hose should not exceed 0.375 in. Stainless steel tubing may also be used (0.25 to 0.375-in. outside diameter), although it is less convenient. Convoluted, flexible stainless steel hose is specifically excluded due to potential entrapment and contamination problems caused by the internal convolutions.

6.3 Condenser—A sample condenser configuration with two sets of stainless steel tubing coils is recommended. One set of coils is dedicated for condensing steam and the other is dedicated for cooling liquid. The steam condenser coil has a pressure/vacuum gage located at the sample outlet and a regulating valve at the inlet. The steam flow can be precisely regulated at the inlet as opposed to regulating the flow of condensate and gas at the outlet that can result in large pressure surges and the hold-up of gas or condensate phases in the coils. The liquid cooling coil has a regulating valve at the outlet and an optional pressure gage. Regulating the outlet flow prevents flashing of liquid at the inlet to the condenser where chemical deposition could occur. Dedicated condensers with single sets of tubing coils for sampling either steam or liquid also can be used (see Fig. 3 and Fig. 4).

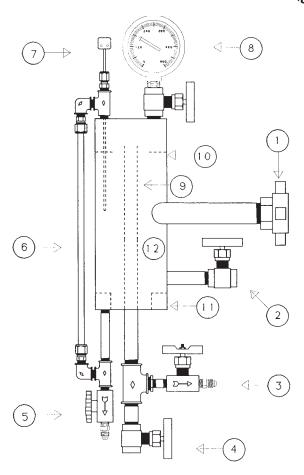
6.3.1 The condenser coil tubing must not exceed 0.25-in. outside diameter to prevent the segregation of gas and condensate phases during sampling of steam. Larger tubing sizes also

increase the risk of contamination and chemical deposition during liquid sampling due to low fluid velocities and longer residence times within the tubing. In cases where the liquid contains substantial quantities of particulate matter, 0.375-in. outside diameter tubing coils may be used to minimize cooling coil plugging problems.

6.3.2 In cases where the noncondensible gas concentration in steam exceeds approximately 5 % by weight, the outlet of the steam condenser coil should be at an elevation below the inlet with a continuous down-slope in the tubing from inlet to outlet. This allows the small volume of condensate to freely drain out of the condenser and prevents hold-up within the coils.

6.4 Condenser cooling can be achieved by an ice/water bath surrounding the coils or by a continuous overflow of cooling water running into the vessel holding the coils (configuration shown in Fig. 3 and Fig. 4). Alternate configurations may include a water-tight jacket around the coils through which a constant source of cooling water flows. A source of coolant may be a glycol/water mixture circulated through the condenser jacket and an external fan-cooled heat exchanger.

6.5 Pressure Gage—For the measurement of separator pressure. Bourdon-tube type gages or pressure transducers may be used. A pressure-snubbing device is recommended to minimize the pressure spikes and surges common in two-phase flow lines. The full-scale pressure range of the gage should not exceed two times the measurement reading. The gage should be calibrated at monthly intervals when in routine use and every six months for intermittent use. The measurement accuracy of the gage should be at least  $\pm 1$  % of full-scale. All



- 1) 1 in. Two-Phase Inlet (Hammer Union)
- 2)  $1\!\!/\!_2$  in. Vent Valve (Regulating Valve or Ball Valve)
- 3) 1/4 in. Steam Sample Valve (Regulating Valve)
- 4) 1/2 in. Steam Bleed Valve (Regulating Valve)
- 5)  $^{1}\!\!/_{\!\!4}$  in. or  $^{3}\!\!/_{\!\!6}$  in. Liquid Sample Valve (Ball Valve)
- 6) % in. Teflon Sight Glass (250 psi limit: 1/16 in. wall, Teflon PFA)
- 7) 1/4 in. × 12 in. Type K Thermocouple
- 8) Pressure Gage with Surge Protector Valve
- 9) ½ in. × Steam Outlet Pipe
- 10) Baffle Ring
- 11) Vortex Breaker Plates
- 12) Separator Body, 4 in. I.D.  $\times$  12 in.

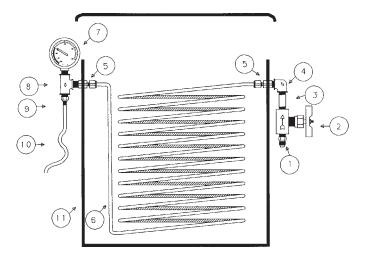
Material specification: All metal components 304 or 316 stainless steel

FIG. 2 Sampling Separator

gages require permanent identification numbers so that field data and calibration data can be traced to each specific instrument.

6.6 Temperature Meter and Thermocouple Probes—For the measurement of separator temperature. Temperature meters are the digital readout style with plug-in thermocouple probes. Type K thermocouples are preferred due to the corrosion resistance of the exposed electrical connectors. The meter accuracy should be at least  $\pm 1^{\circ}$ F. Meters and thermocouple probes should be calibrated at the same intervals as the pressure gages to ensure consistency between the measurements of pressure and temperature. All meters and probes require permanent identification numbers so that field data and calibration data can be traced to each specific instrument.

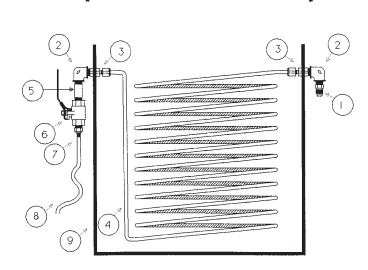
6.7 Fittings—Sample ports often need to be replumbed, and fittings may need to be replaced. A selection of pipe fittings



- 1) JIC Fitting (1/4 in. NPT × S.A.E. 37°)
- 2) Regulating Valve (1/4 in. NPT)
- 3) Pipe Nipple (1/4 in. NPT)
- 4) Pipe Elbow (1/4 in. NPT)
- 5) Bulkhead Fitting (1/4 in. NPT × 1/4 in. Swagelok)
- 6) 20 ft.  $\times$  1/4 in. O.D. Stainless Steel Tubing (0.035 in. wall)
- 7) 30 in. Hg × 30 psi Vacuum/Pressure Gage
- 8) Gage Tee (1/4in. NPT)
- 9) Hose Adapter (1/4in. NPT × 1/4in. Hosebarb)
- 10) Plastic<sup>5</sup> Tubing (% in. O.D., ¾16in. I.D.)
- 11) 8- to 20-Gallon Drum with Lid

Material specification: All metal components 304 or 316 stainless steel

FIG. 3 Steam Sample Condenser



- 1) JIC Fitting (1/4 in. NPT  $\times$  S.A.E. 37°)
- 2) Pipe Elbow (1/4 in. NPT)
- 3) Bulkhead Fitting (1/4 in. NPT × 1/4 in. or 3/8 in. Swagelok)
- 4) 20 ft.  $\times$  ½ in. or ½ in. O.D. Stainless Steel Tubing (0.035 in. wall)
- 5) Pipe Nipple (3 in. × 1/4 in. NPT)
- 6) Sample Valve (Ball Valve, 1/4 in. NPT)
- 7) Hose Adapter (1/4 in. NPT × 1/4 in. Hosebarb)
- 8) Plastic<sup>5</sup> Tubing (3/8 in. O.D., 3/16 in. I.D.)
- 9) 8- to 20-Gallon Drum with Lid

Material specification: All metal components 304 or 316 stainless steel

FIG. 4 Liquid Sample Cooler

including reducer bushings, pipe nipples, couplings and elbows, plus those needed for sample equipment maintenance, are required on site.



- 6.8 *Plastic*<sup>4</sup>— Sample Tubing Located at the exit of the condenser for gas and liquid sample collection (¾16-in. inside diameter by ¾8-in. outside diameter). Heavy wall plastic<sup>4</sup> tubing is required for use under vacuum during gas sample collection and pressure during in-line filtration of liquid samples.
- 6.9 Filter Holder—High-pressure stainless steel (47 mm diameter) with 0.45 micron or smaller membrane filters for use at the condenser inlet. Polycarbonate membranes are required at high temperatures (up to 300°F). The same filter holder can be used at the outlet side or a low-pressure plastic filter holder may be substituted.
- 6.10 *Tool Kit*—Includes at a minimum several pipe wrenches (sizes 24-, 18-, and 14-in.), crescent wrenches (sizes 8- and 12-in.), and a hammer.

#### 7. Hazards

- 7.1 Hydrogen sulfide gas is present in most geothermal steam. Typically the steam itself and the surrounding air are sufficient to dilute  $H_2S$  below hazardous levels. For wells that produce high concentrations of  $H_2S$  (>500 ppm $_{\rm w}$  in steam), a personal  $H_2S$  air monitor is required to verify that safe ambient air levels of  $H_2S$  are not exceeded. This is very important during sampling of shut-in wells or wells on bleed flows which tend to build high gas concentrations.
- 7.2 Hydrogen sulfide gas is concentrated during sampling as the steam is condensed to a liquid. Care must be taken when sampling high-gas wells to make sure that the gas is allowed to dissipate. If this sampling is done in an enclosed building or confined area, a properly sized fan or hood must be used. A personal  $H_2S$  air monitor is also required.
- 7.3 Geothermal steam or water temperatures can exceed 450°F. Dry superheated steam is generally less of a hazard than saturated steam and boiling water. Leather gloves must be worn when working on hot piping, sample hoses, fittings and valves.
- 7.4 Use extreme caution when attempting to vent geothermal fluids through sample ports to the atmosphere. Ports may be plugged with scale or rocks which can be released explosively and unexpectedly after the sample valve has been opened. Warn any assistants or workers nearby to stand clear.
- 7.5 Sample port valves and associated plumbing can be severely corroded or eroded. Extra care must be taken when operating old valves. If the valves are frozen or operate abnormally, they must not be used. Sample port fittings may be too weak to support the weight of the sampling separator. Schedule 80 sample port piping is the minimum grade needed to attach the separator.
- 7.5.1 Do not force valves or fittings with large pipe wrenches. Ruptures and uncontrolled steam releases may result. When tightening fittings with a pipe wrench, verify that the pipe wrench has a "bite" before exerting force and make sure footing is secure.
- <sup>4</sup> Tygon, a registered trademark product of Norton Co., has been used for this purpose.

- 7.6 The noise from geothermal steam bleeds may be above the safe working levels. Ear plugs should be worn during operation of the separator.
- 7.7 Goggles or a face shield must be worn during sampling. An eye hazard exists from debris entrained in venting geothermal fluid and from corrosive chemicals used in sample bottles.
- 7.8 Gas bottles usually contain sodium hydroxide that is hazardous to the skin and eyes. A face shield and eye goggles must be worn when sampling. Gas bottles also may contain cadmium chloride which is poisonous and carcinogenic. Latex gloves should also be worn when handling gas sample bottles.

# 8. Procedure

- 8.1 Installation and Set-Up of the Sampling Separator:
- 8.1.1 Install a Schedule 80, 1-in. pipe nipple, with one part of the connecting hammer union attached to the sample port. Use an elbow fitting and second nipple if the port is vertical. Nipples should be only 4 to 6 in. long. At least a 1-in. size port is needed to support the weight of the separator and to allow sufficient flow without a pressure drop through the separator.
- 8.1.2 Orient the nipple so that it is pointing away from personnel and equipment, downwind if possible. Verify that the path is clear, then open the sample valve to blow out any debris. A qualitative check on the amount of liquid and steam discharged may be made to verify your choice of sample port location. Close the valve.
- 8.1.3 Connect the separator to the 1-in. pipe. Use the hammer to secure the union tightly. Insulate the attached inlet piping and fittings.
- 8.1.4 Close all valves on the separator except the isolation valve to the pressure gage.
- 8.1.5 Open the sample port valve completely and record the pressure reading. This pressure represents the static line pressure.
- 8.1.6 Slowly open the ½-in. vent valve (No. 2 in Fig. 2). Open the valve until the pressure on the gage starts to drop, then shut the valve slightly so that the pressure is maintained at the original static line pressure. Maximize the vent without significantly dropping the pressure (<2 % of reading), so that the relative heat loss between the pipeline and the separator is minimized.
  - 8.2 Operation of the Sampling Separator to Sample Steam:
- 8.2.1 Open the steam bleed valve (No. 4 in Fig. 2) one-half to one turn. Adjust the ½-in. vent valve as needed to maintain static line pressure. Check the sight glass (if available) to verify that the liquid level is lower than the separator inlet. Let the separator level, pressure and temperature stabilize for 5 min. A transparent or bluish steam vent indicates that dry steam is exiting the steam bleed valve. Excess moisture is usually visible as white spray or streaks in the steam plume.
- 8.2.2 Open the steam sample valve (No. 3 in Fig. 2). Allow the steam to blow out any impurities, close the valve and connect the dedicated steam sample hose to this valve. Connect the other end of the hose to the steam condenser coil.
- 8.2.3 Open the steam sample valve 100 % and throttle the sample flow using the regulating valve on the condenser. Adjust the ½-in. vent valve as needed to maintain static line pressure. Allow the condensate to flush for 5 min through the condenser before sampling. Collect all samples as needed

using the referenced methods. Record the separator pressure and temperature before and after collecting samples and monitor during sampling.

- 8.2.4 Prior to and during steam sampling, the separator liquid level should remain below the separator inlet pipe. If this level rises, reduce the steam bleed and steam sample flow rates and open the ½-in. vent valve as much as possible without allowing a significant pressure drop. If the level still rises, another sample port location may be required.
- 8.2.5 If the optional sight glass is not installed, excessive liquid level can be observed when white spray or streaks are present in the steam plume from the steam bleed valve. A surging flow (alternating hissing sound) from the ½-in. vent valve usually indicates that both liquid and steam are venting and the liquid level is at the ½-in. vent valve. A transparent or bluish steam vent indicates dry steam is exiting the steam bleed valve and the liquid level is not too high.
  - 8.3 Operation of the Sampling Separator to Sample Liquid:
- 8.3.1 Open the steam bleed valve (No. 4 on Fig. 2) 100 %. Adjust the ½-in. vent valve as needed to maintain static line pressure. The separator liquid level should rise and may flood the separator, venting liquid and steam from the steam bleed valve. This ensures that pure liquid will be obtained from the liquid sample valve. Let the separator level, pressure and temperature stabilize for 5 min.
- 8.3.2 If liquid is scarce at the chosen sample port, both the steam bleed and steam sample valves may be opened (while maintaining line pressure) and the ½-in. vent valve shut completely in order to flood the separator. Let the separator level, pressure and temperature stabilize for 5 min.
- 8.3.3 Open the liquid sample valve (No. 5 on Fig. 2). Allow the water to blow out any impurities that may have settled there. Close the valve and connect one end of the dedicated liquid sample hose to the valve. Connect the other end to the liquid cooling coil.
- 8.3.4 Open the liquid sample valve 100 % and throttle the sample flow using the liquid sample cooler valve. Adjust the ½-in. vent valve or the steam bleed/sample valves, or both, as needed to maintain static line pressure. Allow the condenser to flush for 5 min before collecting samples. Collect all samples as needed using the referenced methods. Record the separator pressure and temperature before and after sampling and monitor during sampling.
- 8.3.5 Prior to and during liquid sampling, the separator liquid level should remain at or above the ½-in. vent valve as observed on the sight glass. If a sight glass is not installed, the separator should remain flooded as evidenced by liquid present in the steam bleed. If the level falls, operate the separator as described in 8.3.2. The liquid sample rate may also have to be reduced. If the level still falls, another sample port location may be required.
  - 8.4 Collection of Noncondensible Gas Samples:
- 8.4.1 Stem-Type Gas Bottle—The term "stem-type" refers to the type of inlet system used on the gas bottle to collect the sample. A typical stem-type gas bottle is shown in Fig. 5. Stems are usually 5 in. long, having a small hole in the side 3 in. down from the top inlet. The end of the stem tube inside the bottle is sealed shut. The stem is secured to the bottle by a

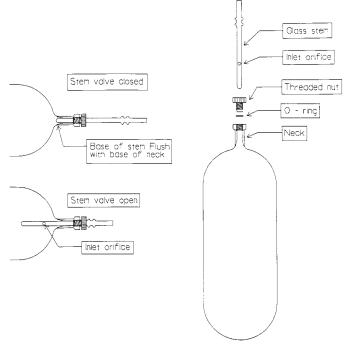


FIG. 5 Stem Type Gas Bottle

threaded packing nut with silicon and O-rings.<sup>5</sup> Tightening the packing nut compresses the O-ring which holds the stem in place with the side hole above the O-rings, maintaining the bottle vacuum. The stem is ridged near the inlet to allow a firm grip with fingers and to prevent it from being sucked completely into the bottle. The sealed end of the stem is enlarged slightly to impede blowout through the O-rings under pressure. Obtain a sample as follows:

- 8.4.1.1 Place the discharge end of the plastic<sup>4</sup> tubing from the condenser gage tee over the end of the stem ½ to ½-in., using care not to push the stem in or twist and break it. Make sure the condenser valve is throttled sufficiently, so as not to over-pressure the tubing. The pressure gage on the outlet of the condenser should not read greater than 15 psig to start. If it does, turn down the sample flow rate. Remember the stem purge pressure at this point; this is the reference pressure when filling the bottle.
- 8.4.1.2 Condensate and noncondensible gas will purge the stem and escape to the atmosphere past the packing nut. As long as the small hole in the stem is above the O-rings, condensate and gas will purge to the atmosphere. When the stem is inserted below the O-rings, the sample is allowed into the container. Gently rotate the stem within the O-rings to loosen and slide the stem into the bottle. Loosen the packing nut one-eighth turn only if the stem does not slide easily.
- 8.4.1.3 After stem insertion, the bottle is turned stem down so that gases and liquids mix. The gage on the condenser should show an increase in vacuum or a drop in pressure, and the sample rate should increase slightly. Due to the small side-hole restriction in the stem, the condenser gage does not

<sup>&</sup>lt;sup>5</sup> Viton O-rings, available from Ace Glass, Inc., 1430 Northwest Blvd., Vineland, NJ 08360, have been found sutiable for this purpose.



read the actual bottle pressure, but rather a relative pressure given the sample flow rate.

- (1) During sampling, the bottle inlet pressure may rise sharply if particulate matter enters the stem and plugs the side-hole. Withdraw the stem before the pressure exceeds 15 psig and try to dislodge the particle, then resume sampling.
- (2) It is desirable to maintain the bottle inlet pressure above atmospheric when sampling by adjusting the sample flow rate. This eliminates the potential for in-leakage of air through the sampling train.
- 8.4.1.4 As sampling progresses, condensate and gases fill the container and the vacuum will slowly decrease back to zero or the pressure will increase back to the initial stem purge pressure. Gently shake the bottle to facilitate absorption of  $\rm CO_2$ . When the initial stem purge pressure is reached, sampling is completed, and the stem should be withdrawn. Slide the stem back up until the side-hole is above the O-rings and condensate/gas is purging again to the atmosphere. The bottom end of the stem should be at the shoulder of the bottle neck, about 1/2-in. below the O-rings. Tighten the nut firmly.
- 8.4.1.5 If the vacuum does not return to zero or the pressure does not increase to the original stem purge pressure, fill the container to approximately three-fourth full with total liquid, but no more than three-fourth full. Those wells producing larger quantities of gas relative to steam will pressurize the bottles much faster and with less liquid volume.
- (1) Although it may be possible to fill the bottle more than three-fourth full with liquid, do not fill bottles beyond this point, regardless of the vacuum remaining in the bottle.
- 8.4.2 Single Stopcock Valve Gas Bottle—Single stopcock valve gas bottles have a sampling nipple to attach the plastic<sup>4</sup> tubing directly to a valve mounted on the bottle. No initial purge is possible—the nipple has a minimal dead volume. A typical stopcock valve gas bottle is shown in Fig. 6.
- 8.4.2.1 Place the discharge end of the plastic<sup>4</sup> tubing from the condenser gage tee over the end of the nipple ½ to ½in. Make sure the regulating valve on the condenser is throttled sufficiently to not over-pressure the tubing. The pressure gage on the outlet of the condenser will continue to increase until the stopcock valve is opened. If the sample rate is too high or if the valve is not opened quickly enough, the plastic<sup>4</sup> tubing will pop off the condenser or the bottle nipple. This usually occurs at approximately 30 psig.
- 8.4.2.2 Upon opening the stopcock valve, condensate and non-condensible gas will rapidly enter the bottle. The vacuum gage will show an increase in vacuum, usually much more than the stem—type bottle due to the larger opening of the stopcock valves. The stopcock valve can be throttled to reduce the sample rate and decrease the inlet vacuum.
- 8.4.2.3 It is desirable to maintain the bottle inlet pressure above atmospheric when sampling by adjusting the sample flow rate. This eliminates the potential for in-leakage of air through the sampling train.
- 8.4.2.4 Invert the container valve down to allow the gas and liquid to mix. Gently shaking the bottle will help dissolve  $\mathrm{CO}_2$  in the liquid and allow more sample to enter the bottle before the pressure increases.

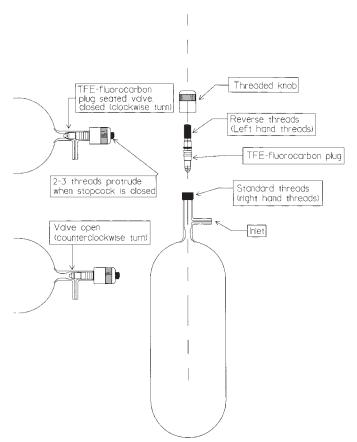


FIG. 6 Stopcock Valve Gas Bottle

- (1) If the sample flow rate is too low, the absorbing solution in the bottle may back-flow into the plastic<sup>4</sup> tubing when the bottle is inverted. Increase flow or keep the bottle upright.
- 8.4.2.5 As sampling progresses, condensate and gases fill the container. The vacuum will slowly decrease back to zero and then the bottle will reach a slight positive pressure. Sampling is completed at this point, and the stopcock valve should be closed. If the vacuum does not return to zero or positive pressure, fill the container approximately three-fourths full with total liquid, but no more than three-fourths full. Slide the plastic tubing off the nipple immediately after the bottle valve is closed. Those wells producing larger quantities of gas relative to steam will pressurize the bottles much faster and with less liquid volume.
- 8.5 Collection of Steam Condensate and Geothermal Liquid Samples:
- 8.5.1 Steam Condensate (see Table 1)—Steam condensate is collected in polyethylene, polypropylene, TFE-fluorocarbon, or glass bottles. Sample bottles not containing preservatives are rinsed at least twice with sample and the end of the plastic<sup>4</sup> tubing is rinsed with condensate from the bottle. The cap is rinsed twice with condensate before tightening onto the bottle.
- 8.5.1.1 If acid or other preservatives are present in the bottles, care must be taken not to spill or overfill the bottles. The condensate temperature should not exceed 80°F during collection, minimizing loss of dissolved gases, and water vapor.



**TABLE 1 Steam Sample Collection** 

Sample Type	Parameter
Gas bottle, evacuated glass containing NaOH/CdCl <sub>2</sub>	Ar, O <sub>2</sub> , N <sub>2</sub> , CH <sub>4</sub> , H <sub>2</sub> , He, CO <sub>2</sub> , H <sub>2</sub> S, NH <sub>3</sub> , saturated hydrocarbons, radon-222, freon and SF <sub>6</sub> tracers
Gas bottle, evacuated glass containing deionized water/CdCl <sub>2</sub>	Unsaturated hydrocarbons, aromatic hydrocarbons, CO
Impinger, gas scrubbing bottle containing NaOH/CdCl <sub>2</sub> ; 0.1 N H <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> S, NH <sub>3</sub>
Raw, unacidified (RU) condensate in polyethylene bottle, 250–500 mL	Cl <sup>-</sup> , F <sup>-</sup> , B, SiO <sub>2</sub> , Na, As, Se
Raw, acidified HNO <sub>3</sub> (RA) or filtered acidified HNO <sub>3</sub> (FA) in polyethylene or TFE-fluorocarbon bottle, 250 mL + 5 mL HNO <sub>3</sub>	Fe, Mn, other heavy metals
Raw, HNO <sub>3</sub> /K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> preserved in glass bottle, 125 to 250 mL + 25 to 50 mL preservative solution	Hg
Raw, unacidified (RU) condensate in glass vial containing copper wire, 25 to 50 mL	Stable isotopes

- 8.5.2 *Geothermal Liquid* (see Table 2)—Liquid sample bottles are filled in the same manner as steam condensate sample bottles.
- 8.5.3 Liquid samples are normally filtered for silica and metals analysis. The filter holder is assembled with a 0.45 µm or smaller membrane filter. Attach the filter holder directly to the discharge end of the plastic<sup>8</sup> tubing from the liquid cooling coils. Reduce the sample flow rate as needed to prevent the tubing from popping off under pressure. Verify that the filter has not ruptured during sampling by inspecting the membrane after the samples have been collected.
- 8.5.3.1 In cases where the suspended matter to be removed may dissolve upon cooling through the coils (calcite) or where precipitates may form upon cooling, the high-pressure stainless steel filter holder can be installed directly at the inlet to the coils using stainless steel JIC fittings. Only polycarbonate membrane filters should be used on the high temperature side.

TABLE 2 Geothermal Liquid Sample Collection

Sample Type	Parameter
Sample Type	Farameter
Raw, unacidified (RU) brine in polyethylene bottle, 500 to 1000 mL	Cl <sup>-</sup> , F <sup>-</sup> , B, SO <sub>4</sub> = , TDS, TSS, pH, alkalinity, As, Se
Raw, acidified HNO <sub>3</sub> (RA) or filtered acidified HNO <sub>3</sub> (FA) in polyethylene bottle, 250 mL, 5 mL HNO <sub>3</sub>	Na, K, Ca, Mg, Sr, Li, SiO <sub>2</sub> (by ICP), Fe, Mn, other heavy metals
Filtered, acidified,~\(^1\)\(^	SiO <sub>2</sub> (by ICP), other high-level metals
Raw, NaOH/CdCl <sub>2</sub> preserved in polyethylene bottle, 500 to 1000 mL, 25 mL preservative solution	H <sub>2</sub> S, SO <sub>4</sub> =
Raw, HNO <sub>3</sub> /K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> preserved in glass bottle, 125 to 250 mL, 25 to 50 mL preservative solution	Hg
Raw, unacidified (RU) condensate in glass vial, 125 mL	NH <sub>3</sub> , total inorganic carbon
Raw, unacidified (RU) condensate in glass vial containing copper wire, 25 to 50 mL	Stable isotopes
Filtered, 0.1 N NaOH preserved (~1/10 dilution of sample with NaOH) in polyethylene bottle. 100 to 250 mL	SiO <sub>2</sub>

Verify that the filter has not ruptured during sampling by inspecting the membrane after the samples have been collected.

8.5.4 Dilution and preservation of liquid samples with HNO<sub>3</sub>, deionized water or dilute NaOH may be required for silica analysis. Dilution with HNO<sub>3</sub> or deionized water may also be required for the analysis of metals and other species in highly saline brines. The dilutions are best performed gravimetrically by adding an approximate amount of geothermal liquid directly from the condenser to a pre-weighed sample bottle containing a known amount of preservative. The exact sample volume and dilution factor can be precisely determined later in the laboratory before analysis.

# 9. Quality Control

- 9.1 Separator Operation—Carefully compare static line pressure to the separator pressure to ensure that there is no significant difference. Reduce vent and bleed flows temporarily to check for an increase in separator pressure.
- 9.1.1 Ensure that the maximum allowable flow is being passed through the separator without a pressure drop and that connected inlet piping and fittings are insulated.
- 9.1.2 Compare separator pressure and temperature readings to steam tables to verify proper instrument operation.
- 9.1.3 Be certain that there is no liquid carry-over into steam samples and no steam entrainment into liquid samples by the techniques described in 8.2 and 8.3.
- 9.2 Gas Bottle Sampling—Whenever possible, gas bottle samples are collected with back-pressure at the bottle inlet as described in the procedure, but no more than 10 psig. By collecting samples in this manner, air contamination from condenser fitting leaks can be minimized. Stem bottles usually are best suited for sampling with back-pressure, but stopcock bottles can also be used in this manner by throttling the stopcock valve. The steam condenser should be leak-checked regularly by drawing a 20-in. Hg vacuum on the condenser, shutting off the vacuum source, and verifying the system will maintain this vacuum for 10 min.
- 9.3 Condensate Samples—Properly cleaned and prepared sample bottles are obtained from qualified laboratories and are rinsed with condensate in the field when appropriate. Only condenser trains and sample hoses dedicated strictly for steam sampling are used. Steam samples are not collected with geothermal liquid sampling equipment.
- 9.3.1 Sample train equipment must be sufficiently flushed between wells and upon initial sampling to prevent cross-contamination. Typically, a 5-min condensate flush at full flow is required before any samples are obtained.
- 9.4 Geothermal Liquid Samples—Properly cleaned and prepared sample bottles are obtained from qualified laboratories and are rinsed with liquid in the field when appropriate.
- 9.4.1 Sample train equipment must be sufficiently flushed between wells and upon initial sampling to prevent cross-contamination. Typically, a 5-min liquid flush at full flow is required before any samples are obtained.
- 9.4.2 Check filter membranes after collecting filtered samples to ensure that the filter is intact.
- 9.4.3 Check for excessive gas bubbles in the liquid sample stream which may indicate steam entrainment.



# 10. Keywords

10.1 chemical analysis; fluid chemistry; geothermal resources; two-phase geothermal fluids

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