Title

OPERATING INSTRUCTIONS FOR LBL/GRI DOWNHOLE SAMPLER AND SAMPLE EXTRACTION SYSTEM

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DISCLAIMER

OPERATING INSTRUCTIONS FOR LBL/GRI DOWNHOLE SAMPLER AND SAMPLE EXTRACTION SYSTEM

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1. Introduction

With support from the Gas Research Institute, the Lawrence Berkeley Laboratory has designed, fabricated, and tested a downhole sampler that may be used to bring fluid samples up from geopressured gas wells. Auxiliary tools needed to utilize the full potential of this sampler were also designed and built. This work is described in two annual reports (Michel *et al.* 1982, and Weres *et al.* 1984). The present document contains operating instructions for this equipment, and is an addendum to Weres *et al.* (1984). A full scale cross-sectional drawing of the sampler (LBL drawing no. 47-30-01/18B3815) is a necessary attachment to this document. This drawing will henceforth be referred to as Fig. 1.

The best way to familiarize yourself with the construction and operation of the sampler is to color-in Fig. 1 with felt-tip markers or colored pencils.

The operating instructions in this document were written after the sampler and its auxiliary equipment were tested in the laboratory, but before the sampler was actually used in a geopressured well. Therefore, these instructions are subject to future revision.

2. The sampler

2.1. Principles of operation

The drawing shows the sampler in its "valves open" configuration. The cable is a 7/32" dia. monoconductor cable sheathed with MP35N. Current flowing down the single conductor of the cable keeps the sampler valves open. The magnet assembly is held stationary. The armature subassembly is mobile; it is rigidly attached to the piston rod, arming piston, and upper valve release rod; these parts move together. The piston return spring pushes these parts upward; when the sampler is cocked open, this force is countered by the magnetic attraction between the magnet assembly and the armature subassembly. Note that the current that energizes the magnet coil flows through electrical contacts from the armature subassembly to the magnet assembly. The upper valve spring

pushes the upper valve stem and upper valve coupling upward. This force is transmitted to upper valve arming rod. The upper valve arming rod is prevented from moving by the three lock balls, which lock it to the upper valve ball cage. This coupling (through the lock balls) between the upper valve arming rod and the upper valve ball cage keeps the upper valve open. The lower valve is kept open in exactly the same way.

A small upward flow of water past the sampler is needed to exchange fluid with the wellbore; if necessary, this relative velocity may be provided by lowering the sampler through stationary fluid. Brine flows up through the holes in the bullnose ("bullet-nose" in Fig. 1), through the eight small passageways in the lower valve guide, and into the lower filter chamber (inside the lower transition sleeve). There the brine flows concentrically in through the filter, through the lower valve spring, and up the passageway that parallels the lower valve stem. It flows past the open valve's seat, through the small holes in the lower part of the lower ball cage, and into the bottom of the sample volume inside the pressure vessel body. The brine flows up the length of the pressure vessel body, past the open upper valve's seat, through the passageway that parallels the upper valve stem, and finally out through the upper valve spring, upper filter, and the holes in the intermediate transition sleeve.

Interrupting the current from the surface causes the sampler to close. When the current is interrupted, the magnet releases the armature subassembly, allowing the armature subassembly, the piston rod, the arming piston and the upper valve release rod to be impelled upward by the force of the piston return spring. The upper valve release rod pulls the upper ball release sleeve up with it, until the notches inside the upper ball release sleeve line up with the lock balls. Then the lock balls fall into these notches, releasing the upper valve arming rod. With the upper valve arming rod free to move upward, the upper valve spring pushes the upper valve stem up, causing the upper valve to close, and also pulling up on the lower valve release rod. The lower valve release rod pulls the lower ball release sleeve up until the notches in it line up with the lower three

lock balls. The lock balls release the lower valve arming rod, allowing the lower valve to be pulled down, and thereby closing the lower valve.

Valve closure is irreversible, because separation of the magnet and armature assemblies opens the circuit; closing the switch at the surface is therefore insufficient to reenergize the magnet.

2.2. Operational limits

The sampler is designed to contain fluid when the pressure inside the sampler is higher than the pressure outside. It was engineered safely to contain fluid at pressure up to 25,000 psig. The design temperature of the sampler is 450°F; in fact, it has successfully been operated at 500°F (Michel *et al.*, 1982, Appendix D).

Twenty-five thousand psig is maximum pressure rating of the tool. The practical pressure rating will be determined by the rupture disks that are used. Rupture disks rated 10, 15 or 20 kpsi will be appropriate to most applications.

The sampler's pressure rating applies to the maximum pressure *difference* between its contents and its environment. There is no limit on the *absolute* pressure that the sampler may be subjected to. In principal, the sampler could be used in wells with bottom hole pressure greater than 25,000 psi.

When the valves first close, the pressure inside the sampler will be approximately equal to the pressure outside. When the sampler is then pulled back up the wellbore, the pressure outside the sampler will decrease rapidly, ultimately reaching the static wellhead pressure. In a normally pressured well, the static wellhead pressure will be a few hundred psi or less. In an abnormally pressured (i.e., geopressured) well, the static wellhead pressure may be as high as several thousand psi.

The pressure inside the sampler when it reaches the wellhead will depend on the temperature of the wellbore fluid at the wellhead and the gas content of the sampled fluid. If the wellhead temperature is equal to the tempera-

ture at the point of sampling, the pressure inside the sampler will be approximately equal to the pressure at the sampling point. In this case, the pressure inside the sampler will be much higher than the wellhead pressure. The maximum outward pressure differential will be encountered when the lubricator is depressurized and the sampler is removed from the wellhead. Leakage should not occur if this pressure differential is less than 80% of the nominal rating of the rupture disks. In practice, the sampler is most likely to fail by blowing a rupture disk or extruding an O-ring from one of the valve seats. If the O-ring is defective or improperly installed, failure may occur at a pressure much lower than 25,000 psi.

If the well has not been produced very much recently, the temperature of the wellbore fluid at the wellhead may be substantially lower than the downhole temperature. In this case, the fluid inside the sampler will cool when it is brought up to the wellhead, and the pressure inside the sampler will decrease. If this temperature drop is large, and the well is strongly geopressured, it is possible that the pressure of the wellbore fluid at the wellhead will exceed the pressure inside the sampler. If the inward pressure differential exceeds a few hundred psi, a valve may be forced open, allowing inward leakage of fluid. If the upper valve opens, some gas may escape from the sampler. This is expected to be a very rare situation in practice. This matter has been discussed in detail in Appendix A of Michel *et al.* (1982).

The sampler is designed to sample single phase fluid. If free gas is present in the wellbore fluid, some gas bubbles will be excluded from the sampler, and the proportion of gas to water in the sample collected will be less than in the wellbore fluid.

2.3. Sampler operating instructions

We proceed to describe the series of steps needed to prepare the sampler for a typical sampling run. The sampler is to be clean and fully assem-

bled when you begin.

Remove the cable head from the sampler, and screw the removable stinger assembly on to the cable head. Thread the cable through this subassembly. The cable head incorporates the widely used brass cone cable termination for mechanically attaching the cable to it. Be sure you know what you are doing, or the sampler might be lost downhole; if you don't, ask someone who does to supervise. Refer to Fig. 1 as to how the electrical conductor connection is accomplished.

Screw the cable head on to the sampler.

Mount the cocking tool on the sampler, and push the arming piston and attached parts down with the cocking tool (there are appropriate attachment points on the sampler). Use the power supply/ control unit to energize the magnet as described in the following section. Maintain the current from now on, until ready to take a sample down inside the well.

Cock open the lower valve by screwing the threaded portion of the "T-handle" device into the internally threaded tip of the bullnose. This causes the lower valve spring to compress, forcing the lower valve stem upward, and letting the lock balls engage and hold the lower valve in the open position. The opening of the lower valve has been accomplished when an audible clicking sound is heard. This indicates that the lock balls have been engaged. The pronounced spring pressure on the T-handle that was felt during the cocking operation should not be noticeable during the removal of the T-handle, since the lower spring is now held compressed. If the spring pressure is still felt during the removal of the T-handle, the cocking procedure should be repeated.

Four sinker bars are provided to provide additional weight, if desired. To add additional weight, first unscrew the stinger from the cable head. Remove the cable retaining strip from a sinker bar. Place the sinker bar over the cable. Replace the cable retaining strip and screw it into place; now the cable should run the length of the sinker bar and be enclosed by it. Screw the sinker bar on to the cable head. Repeat these steps if additional sinker bars are to be added.

Finally, screw the stinger on to the top-most sinker bar.

The electrical components of the sampler are immersed in Krytox oil to protect them from the brine. The oil must be pumped into the sampler after it has been suspended from the cable in a vertical position. Once the oil has been put into the sampler, the sampler must remain in a vertical position. The oil is viscous at room temperature, and the oil-filled part of the sampler must be heated to reduce the viscosity of the oil and make it easier to pump.

The oil is put into the sampler as follows. Suspend the sampler from the cable. Fill the oil pump with Krytox oil. Lace the heating mantle on to the sampler. (Heating tape may also be used.) Adjust the heating mantle so that the the oil-fill hole in the sampler is accessible through the hole in the heating mantle. Remove the oil-fill plug, and attach the oil-pump. Connect the heating mantle to a source of 110 volt power, and allow it about 10 minutes to heat up that portion of the sampler to 250^{o}F. (The heating mantle contains thermostats set to this temperature.) Now pump the oil into the sampler.

It takes approximately 250 ml to fill the portion of the sampler above the oil fill hole. Oil should be added until the oil starts extruding from the top of the cablehead. (This may be observed by removing the cable retaining strip of the lowest sinker bar; if no sinker bars are used, temporarily unscrew the stinger from the cable head.) The oil pump barrel has a useful volume of approximately 80 ml; therfore, three pumpfulls will provide the volume needed. Any unused oil should be removed from the pump and saved for later use. The sampler oil should be topped-off before each subsequent run into a well or if the sampler is placed in a horizontal position after the oil filling has been completed.

When done, disconnect the pump, replace the oil fill-hole plug, and remove the mantle.

The sampler is now ready to enter the wellbore.

The brine may contain suspended particles large enough to interfere with proper sealing of the sampler's valves. The sampler has brine filters that prevent this from happening. Normally, these brine filters consist of 100 mesh

nickel screen, fastened to filter cores of MP35N with nickel wire. If the brine contains a large amount of suspended solids, enough solid material may accumulate on the lower filter to interfere with the flow of fluid through it. If this is suspected, the lower brine filter may be cleaned in the wellbore. After the sampler has reached sampling depth, quickly retrieve it about 200 feet. Reversing the flow of brine through the sampler in this way should dislodge any solid particles caked on the filter. Then lower the sampler back to the desired sampling depth and take the sample. This final descent of about 200 feet will allow a fresh, representative fluid sample to be taken.

The sampler valves are closed by cutting off the cable current as described in the following section. Removal of fluids from the sampler is described in the second section following.

2.4. Power supply and control unit

The power supply/ controller provides the current to the sampler magnet needed to keep the sampler valves open while the sampler is being lowered to the desired depth. The controller is normally operated on 110 volt AC power. It also has rechargeable back-up batteries, which will keep it operational in the event of an AC power failure.

The controller is designed to provide a specified flow of current through the conductor of the cable to the sampler. It is equipped with digital readouts for the current and voltage through the cable + sampler magnet. The current readout is used to adjust the current provided to the sampler, and gives an indication of valve closure; the current goes to zero when the sampler valves close.

In principle, the voltage and current readouts may be used to estimate the temperature profile in the wellbore. First, you would have to measure in the laboratory the resistance of the magnet coil and the resistance of the cable as a function of temperature. The temperature as a function of depth could then be calculated from the resistance *vs.* depth data collected in the field.

CAUTION

External monitoring equipment may be connected to the controller. The controller has connectors for this purpose. To avoid potential ground loops and accidental valve closure, connect only differential inputs, and only to these connectors. Do not connect external equipment directly to the sampler lines.

2.4.1. Power supply specifications

1. Current controlled supply with continuously adjustable range of 0-50 mA, equipped with a multiturn indicating knob.
2. Maximum output voltage approx 48 VDC at 50 mA.
3. Output current and voltage read on independent built-in digital meters. Each meter reads four significant digits and is protected against overload.
4. Output connections provided for external monitoring of current and voltage.
5. The power supply operates from 120 VAC and contains standby batteries that will provide power for 12 hours during absence of primary power.
6. The power supply is protected against:

a) AC line noise and spikes

b) Noise and inductive spikes from the downhole cable, sampler magnet and contacts. The protection goal is to maintain current through the sampler magnet.

7. Key protected switches to prevent accidental release.
8. AC Power Indication.
9. A protected DC polarity reversal switch is provided to reverse output voltage polarity for cancelling residual magnetism and providing additional kick during release.
10. Fusing and other protective measures designed so as not to interrupt power to the sampler if at all possible.
11. Use of high-reliability components.

12. Use secure mechanical construction to defend against rigors of field use and transportation.

2.4.2. Charging batteries

1. Turn CURRENT ADJUST fully CCW.
2. Connect unit to 110 VAC.
3. Turn AC Power and BATTERY switches on.
4. Observe that AC Power On light is lit.
5. Batteries will achieve full charge in 24 hours.

2.4.3. Testing unit

1. Turn CURRENT ADJUST fully CCW.
2. Connect unit to 110 VAC.
3. Observe that the panel meters and AC Power light are lit.
4. Place the LOAD switch in the DUMMY LOAD position.
5. Plug the DUMMY LOAD plug into the OUTPUT receptacle.
6. Turn AC and BATTERY Power switches on.
7. Set CURRENT ADJUST for a reading of 40.00 (mA) on the MILLIAMPS panel meter.
8. Turn the AC Power switch off. Observe that the current reading does not drop more than 0.02 mA.
9. Turn the AC Power switch on and the BATTERY switch off. Observe that the current reading does not drop more than 0.02 mA.
10. Turn AC Power and BATTERY switches off.

2.4.4. Energizing sampler magnet

1. Perform the Test Procedure above to check unit and set current to the required 40 mA.

2. Connect cable from sampler to output receptacle.
3. Turn on both AC and BATTERY Power.
4. Cock the Tool. Observe the milliameter reading when the contacts in the sampler close during cocking.
5. Adjust current to 40.00 mA (or other holding value).
6. Observe that the voltage is less than about 40 volts.
7. Turn the AC Power switch off and observe that the current reading does not drop more than 0.1 mA.
8. Turn the AC Power switch on and the BATTERY Power switch off and observe that the current does not drop more than 0.1 mA.
9. Turn the BATTERY Power switch on.
10. Remove both AC and BATTERY Power switch keys to insure against accidental release of the sampler solenoid.

2.4.5. Closing the sampler valves

1. Turn Current Adjust fully CCW slowly, observing voltage and current on the panel meters. When the magnet drops out, closing the sampler valves, the current will suddenly drop and the voltage suddenly rise.

   When dropout occurs proceed to step 6.
2. If the full CCW position was reached and the magnet did not appear to drop out rotate the CURRENT ADJUST CW 2 turns and observe the current and voltage. If the current is zero and the voltage is high the sampler has dropped.
3. If the sampler has still not dropped return the CURRENT ADJUST to full CCW position and release the POLARITY REVERSAL switch by removing the pin from the switch guard.
4. Place the POLARITY REVERSAL switch in the REVERSE position and slowly advance the CURRENT ADJUST, observing the voltage and current for the dropout indication.

5. If dropout has not occurred and 40 mA is again reached try reversing the polarity again, perhaps faster. Go to step 3.

6. Turn CURRENT ADJUST fully CCW, POLARITY REVERSAL to NORMAL, replace locking pin on POLARITY REVERSAL. Install keys and turn off both AC and BATTERY Power switches.

2.5. Field maintenance of the sampler

After the sample has been removed from the sampler, the sampler must be partially disassembled, cleaned and inspected, and reassembled. The two rupture discs must be replaced, the valve mechanisms must be cleaned and inspected, and the working volume must be cleaned and made ready for the next sample.

Remember to have sufficient rupture disks and Krytox oil and grease on hand; two rupture disks and up to 250 mls of Krytox oil will be consumed on each sampling run.

A variety of Inconel rupture disks are included with the sampler, with room temperature pressure ratings from 5.5 to 28 kpsi. Their yield strength will be 10-25% less at elevated temperature. These are stock rupture disks, with 3/16" diameter faces, and 3/8" outer diameter. Some of them have been coated with Teflon for greater corrosion resistance.

The sampler's major external parts are shown in Fig. 2, which illustrates how the section pairs are identified at their parting lines. The figure indicates the limit of disassembly for normal field maintenance, as well. Also, refer again to Fig. 1.

2.5.1. General comments

Work on the sampler thoughtfully and systematically. Don't lose any of the parts. There should be storage places for the parts as they are removed from the sampler. There should be enough wooden "vee" blocks to hold various the cylindrical pieces to prevent their rolling about. There should be a good bench,

and there should be a clean floor so dropped parts can be found. There should be time available to consider the process, and there should be plenty of light for complete and thorough inspection.

Included with the sampler tools is a container of Krytox grease mixed with tungsten disulfide. At reassembly, the cleaned and inspected thread systems should have a fresh coat of this mixture applied. A stiff Nylon toothbrush will work well as an applicator. Note that MP35N has a strong tendency to gall; to disassemble seized parts may require considerable time and ingenuity and it will pay to take care at the assembly time.

One section of the sampler cannot be unscrewed from the next without first removing a Lockscrew (18B3762) from the sections pair's parting line. Reassembly of any section pair requires good alignment of the two halves of the tapped set screw hole.

2.5.2. Rupture disc replacement

There are two rupture discs, one located in either of the two Valve Couplings (18B3532). The Valve Couplings are located at opposite ends of the pressure vessel, inside the Lower Valve Guide (18B3793) and inside the Upper Valve Guide(18B3563). Note the Valve couplings are identical, but the Valve Guides differ. The Rupture Disc is held against a Hold Down Ring(18B3542) seated into a counterbore within the Valve Coupling, by the Lower Valve Stem(18B3573-A) or the Upper Valve Stem (18B3573-B). The Valve Stems are threaded into the Valve coupling.

Replacing a disc is a similar sequence for either location. In the following example, the Lower Disc will be replaced. It is assumed the tool's "lower section", up to joint H-H, as indicated in Fig. 2, is resting in "vee" blocks with the Bullet Nose (18B3803) to the operator's right.

PROCEED:

1. Disengage Lockscrew (18B3762) at Joint A-A.

2. Unscrew Bullet Nose (18B3803) from Lower Valve Guide (18B3793) at Joint A-A.
3. Disengage Lockscrew at Joint B-B.
4. Remove Valve Guide Pin (18B3552).
5. Unscrew Lower Valve Guide from Lower Transition Sleeve (18B3772). The Valve Coupling is now exposed. Also, the Filter Assembly (18B3623) can now be removed and cleaned.
6. Remove the Valve Plug (18B3522).
7. Back off the Valve Stem Nut (18B3602) from the Valve Coupling. (There is a wrench flat on the Valve Stem).
8. Unscrew the Valve Coupling from the Valve Stem.
9. The Rupture Disc and the Hold Down Ring can now be removed from inside their coupling.

   It might be necessary to insert a 5/32" drift punch, in from the right end of the Coupling, to help dislodge the Disc and the Hold Down Ring.
10. Clean all parts.
11. Reassembly is the reverse of the procedure just described.

NOTE: The Hold Down Ring has a slight radius on one end of its bore; the disc must seat on that radius, and the disc bulge must be entering the ring's bore. Be sure the parts are in their proper places when they are assembled.

If the tool is to be cleaned and inspected, do not reassemble the Disc and Ring assembly at this time.

If the tool is to be reassembled at this time, the Valve Coupling and the Valve Stem should be assembled and torqued to 60 foot lbs. The "jam" nut (Valve Stem Nut, 18B3602) should be torqued to about 10 foot lbs.

2.5.3. Further disassembly

12. Remove the remaining lockscrews at the various joints along the tool.
13. Remove the Lower Transition Sleeve (18B3772) and the Intermediate Transition Sleeve (18B3632).
14. Remove the Valve Stem Nuts (18B3602) from both the Upper and Lower Valve Stems (4 nuts total).
15. Remove the Valve Spring (18B3781) and Spring Retaining Washer (18B3612) from the upper and lower assemblies.
16. Unscrew the Upper Closure (18B3642-B)
17. The Upper Valve Stem is pinned to the Lower Valve Release Rod (18B3712) which is in turn attached to the Lower Ball Release Sleeve (18B3732).

    It is best to not pull the Upper Valve Stem from the vessel at this time, although it can be done: but to do so will pull the Ball Release Sleeve away from the Ball Cage, and the Lockballs(18B3481) will fall free inside the vessel. Instead:
18. Maintain the Upper Valve Stem's position relative to the Lower Closure. Remove the Pressure Vessel Body (18B3653) from around the Stem Assembly.
19. Unscrew the Lower Closure from the Lower Ball Cage (18B3753).
20. The Lower Ball Release Sleeve can now be withdrawn over the Lock Balls and Ball Cage. The Lock Balls will fall free. (At reassembly, the Balls can be temporarily retained with a rubber band around the Ball Cage. When the Cage is inserted into the Retainer Sleeve, the band will stay behind.)

What remains assembled on the bench includes the Upper Valve Stem (with its 'O' Ring) which is pinned to the Lower Valve Release Rod; The Lower Ball Release Sleeve, which is retained to the Release Rod by a Collar (18B3182) and a pin; and the Lower Valve Arming Spring (18B3721), which is retained by a Collar

and a pin on one end, and by the Lower Release Sleeve at the other end.

The Lower Ball Cage is threaded into the Lower Valve Stem, and is pinned after being installed; this assembly is now also on the bench, and is separated from the Valve Release parts just described.

The parts can now be cleaned and inspected without further disassembly. Further disassembly should be necessary only for repairs and/or replacement of parts.

Closure 'O' rings are retained to the Upper Closure by a Retainer (18B3672) and to the Lower Closure by the Lower Ball Sleeve. Note that each Closure 'O' ring has a Back Up Ring(18B3662). Note that upon reassembly, the Back Up Ring is installed first, then the 'O' ring is installed.

Valve Stem 'O' rings are removed by sliding them along the length of the Stem, and off the smaller end. When installing 'O' rings to this part, use care when sliding them along that they do not become cut, or abraded. It is advisable to lubricate the valve stems with Krytox grease before installing the O-rings.

NOTE: The Upper Ball Cage (18B3472), the Upper Ball Sleeve (18B3462), the Upper Valve Arming Rod (18B3492), and the Arming Rod Retaining Screw(18B4401) are all still attached to the upper section of the sampler that was not disassembled, and in any normal conditions this region should not have to be worked on, except as will now be described:

The general assembly within the release housing is a mechanism which is subjected to the same contaminants and debris as the rest of the sampler's active volume. There are connecting passages drilled through the Valve Guide which will allow passage of a cleaning blast of compressed air or alcohol. There should be no very large particles in this region because all the fluid had to pass first through either the upper or lower filter assemblies. The cleaning suggested here should prevent an eventual build-up of junk that may hinder tool operation at a later time. Also, it will reduce the probability (in any case small) of introducing a contaminant in any future samples.

The reassembly is essentially the reverse of all operations described for

disassembly.

3. The sample extraction system

3.1. Design and function

The sample extraction system (SES) is diagrammed in Figures 3 and 4. Figure 3 shows the "minimum" configuration of the SES. This minimum configuration allows the brine and gas to be removed from the sampler, separated, and packaged for shipment, all without exposure to air. The total amount of gas in the sample is determined by measuring the absolute gas pressure in the SES and gas sampling bottles, the total volume of which is known (see Section 3.6).

Three kinds of brine containers are provided:

(1) Small glass bulbs with integral glass valves are provided in several sizes (K in the Figures). There are a total of eight such bulbs, with volumes ranging from about 25 ml to 150ml. These bulbs provide the best protection against leakage and air contamination, but are small and inconvenient to work with. Because they have only one narrow opening (the valve), vacuum must be used to remove the contents, and cleaning is difficult.

(2) A single large (about one liter) sub-boiling evaporator is provided (H). This vessel is partially filled with brine in the field. In the laboratory, nitrogen may be bubbled through the brine while it is heated gently. This allows the brine to be evaporated without exposure to air. (Evaporated brine is needed for analysis by neutron activation or X-ray fluorescence.) The sub-boiling evaporator may be opened for cleaning.

(3) For routine analysis, brine is collected in modified 250 ml gas washing bottles (I). Four of these containers are provided. Short lengths of Tygon tubing are connected to the hose barbs on the gas washing bottles, and sealed with screw clamps. The bottle is connected to the SES through Tygon tubing that is attached to a valve with a hose barb (8 in the Figures).

A pH electrode is mounted in a special flow-through plastic fixture (L), and attached to the SES through a glass valve. Flowing brine through this fixture allows the pH of fresh brine to be measured before it is exposed to air.

In its "maximum" configuration (Fig. 4), the SES has additional capabilities. The brine may be filtered before it is bottled. A 142 mm diameter membrane filter in a modified stainless steel filter housing is employed (C). The filters supplied with the SES are Millipore type GVWP. They are made of polyvinylidene difluoride, and the pore size is 0.2 μmeters. Glass fiber prefilters (Millipore AP15) are also supplied. These are 124mm in diameter, and have a nominal pore size of 1μmeter.

If so desired, a sample of brine may be acidified and gas stripped with helium, and the gases thus removed collected on molecular seive material. The brine to be gas-stripped is collected in vessel J, which has a septum that allows acid to be injected into it. The gas evolved by this operation is collected in cryotraps F, which contain molecular seive material and are cooled with liquid nitrogen. These cryotraps may also be used to concentrate the less volatile (ethane and heavier) components of the gas in the field.

The design philosophy was to provide all capabilities that might be desired of the SES. Many of parts of the SES are interchangeable, and the configuration of the system is flexible.

Gas and brine containers sufficient for two sets of samples are provided; however, only one sub-boiling evaporator (H) and one gas stripping vessel (J) are included. In practice, the bottles I and gas bottles E are most important. Provide more of these if more than two sets of samples are to be collected.

3.2. Materials of construction

The various parts of the SES are attached to the plywood backboard by threaded metal rods that are bolted to the board. The smaller parts are attached to the rods with plastic cable ties. Support rods for the larger parts (B, C, D, E, H, J) are provided with aeroseal clamps that are padded with vinyl tape and actually

grasp the part. Most connections inside the system are 1/4 inch Cajon VCR's, including metal-metal, glass-glass and glass-metal connections. The gaskets used with the Cajon connectors are made of 1/16 inch Viton sheet. The vacuum pump connection employs 1/2 inch UCLRL fittings. The connections to the sampler and the helium supply are 1/4 inch Swageloks or Gyrolocks.

Throughout most of the SES, fluids contact only glass, Teflon, Viton, and stainless steel (types 304 and 316). Most of the parts that contain liquid are made of glass (the left side in Figures 3 and 4). This makes it possible to see what is going on inside, and expedites cleaning, thereby reducing sample contamination. Glass vacuum valves with Teflon stems and Viton O-rings for seats and seals are used. The flexible braided steel hoses connected to the cyclone separator are lined with Teflon. The brine sampling manifold (G) is welded stainless steel. (A glass manifold is also available if needed, but is too fragile for routine field use.)

The gas handling part of the system is metal. Nupro stainless steel bellows valves are used (Nupro part number SS-4H-TW). In most places Cajon glands are welded to stainless tubing, and most of these welds were made under an inert atmosphere. A few parts (all on the gas side of the system) are silver soldered. The only brass in the system is in the pressure gauges and associated fittings, which contact gas only.

The system was designed to allow glass breakage to be avoided or compensated for. All glass parts were fabricated of heavy walled glass stock and annealled. (For example, the cyclone separator (B), lower brine reservoir (D), and sub-boiling evaporator (H) are made from industrial borosilicate glass pipe.) There are glass spares for all the small glass parts (valves, T's, crosses). Ordinarily, the glass cyclone separator will be used. Another separator, made of 304 stainless steel, is also provided. The steel unit may be substituted if the glass separator is damaged.

The lower brine reservoir (D) is not essential; if it is damaged, it may be replaced by a straight run connector, or by a flexible hose. The sub-boiling evaporator H and gas stripper J are not essential. Non-essential components may

easily be eliminated from the system by blocking-off their connectors with Cajon nuts backed by blank Viton disks.

3.3. System operating requirements

A level area protected from the weather is needed to set up the SES. A work bench or table is needed for parts and tools. A variety of small tools (wrenches, screw drivers, etc.) are needed. A portable vacuum pump that operates on 110 VAC power is provided with the SES. A cylinder of helium or nitrogen with a regulator is needed. A regulator is included in the SES tool kit, but the cylinder must be provided in the field.

If the cryotraps are to be used, the bottled gas must be helium. Also, a Dewar of liquid nitrogen will be needed to cool the cryotraps.

3.4. System assembly

Normally, the SES will be shipped with all glass parts removed and packaged in their wooden shipping case. The heavier metal parts (filter housing, gas sample bottles, cryotraps, pressure gauge assemblies) and some small metal parts that protrude beyond the edge of the backboard must also be removed and packaged for shipment. Most of the metal tubing and valves may be shipped attached to the backboard.

Caution must be exercised while assembling the sample extraction system to avoid glass breakage. Be sure that each glass Cajon gland is backed by an O-ring that cushions the contact between the glass and the metal nut. Be sure that a Viton gasket is in place between the glands before tightening the nuts. Be sure that parts are properly aligned before connecting them. Glass parts *cannot be stressed*, and *will not bend or stretch*. However, metal parts may be distorted slightly during assembly, and this can be exploited to avoid stressing the glass parts. Also, the Viton gaskets confer some flexibility to the joints.

Do not use a wrench on to tighten any of the Cajon connectors. A wrench may damage glass Cajon glands, or cause metal glands to cut through the Viton gaskets.

Begin by tightening all Swagelok fittings with a wrench. If the filter is to be used, mount it first, and properly position all adjacent glass parts working out from the filter housing. It is important that the filter housing be as close to horizontal as possible. If necessary, position it using shims and tape.

While assembling the system, first connect all Cajon connections loosely. Completely assemble the glass part of the system before tightening the connections. Before tightening, verify the absence of stress by disconnecting the connections one at a time, and observing that the glands remain in alignment. That the glands are parallel and 1/16 inch apart is more critical than perfect alignment of their axes. However, axial alignment should be within 1/8 inch as well. The distance from the backboard may be adjusted by screwing the mounting rods in or out as necessary.

The SES is easy to assemble in its minimal configuration. In this case, the only critical alignment is among the lower brine reservoir (D), the valve below it, and the brine sample manifold.

The Viton gaskets should be coated lightly with Krytox grease, as this will reduce the pressure needed to achieve a good seal. The Viton gasket in the cyclone should also be greased lightly. (Vessels D and H have Viton O-rings in their large joints.) Greasing is essential at all glass-glass and glass-metal connections, and useful at metal-metal connections.

After correct alignment has been attained, carefully tighten the Cajon connections, beginning with those along the vertical axis on the left side of the system. *Do not use a wrench*; hard finger tight provides an adequate seal, with little danger of breakage. Adjust part alignment as necessary while tightening the connector nuts. Finally, tighten the metal-metal connectors.

Connect the desired brine sample containers to the manifold. The modified gas washing bottles (I) and the sub-boiling evaporator (H) are connected by Tygon tubing to glass valves that are attached to the manifold. Another piece of Tygon tubing is attached to the gas exhaust of the gas bottle; this is pinched off with a metal tubing clamp.

Before connecting the pH electrode block, flow some standard pH 7 buffer through the block and calibrate the electrode. Be sure the rinse the buffer out with water before attaching the block to the manifold.

Unused positions on the sample manifold and other "open" Cajon connectors in the SES are blocked off. To block the female Cajon connectors on the manifold, use a blank Viton circle backed by a Teflon circle and a male Cajon nut. To block male connectors, use a Viton circle, a Teflon circle, and a small female Cajon nut. Remember to put some Krytox grease on the Viton circle.

Connect the vacuum pump and the helium (or nitrogen) cylinder. Be sure to provide a water trap between the SES and the vacuum pump. A glass filtering flask makes a good trap. If the flask is clean and empty to start, whatever brine accumulates there during SES operation may be saved for analysis. Connect the auxiliary helium line between valve 18 and lower piercing valve M. A armored hose is provided for this purpose.

3.5. Operating instructions

Before trying to use the SES in the field, be sure to assemble it and operate it in the laboratory. Filling the sampler with water that is saturated with CO_2 makes for a safe but realistic excercise. Use the sampler without rupture disks for this test. Use vacuum to pull about 950 mls of water into the sampler. Connect the lower valve to a CO_2 cylinder regulated to 90 psi, and let the gas flow through the sampler and escape from the upper valve for a few minutes. This will provide a gas: water ratio of about 20-30 SCF/bbl.

The detailed operating procedures will depend on the exact configuration of the SES, and, in many cases, alternative procedures may be employed. Strict adherence to procedure is essential only where safety is concerned. These points are emphasized in the text. Detailed operating instructions are given for the maximal configuration of the SES only.

Rather than following the instructions presented here by rote, think about what you are trying to do, and what valve settings are needed to accom-

plish it.

Refer to Figure 4. Assemble the sample extraction system as described above.

Attaching the sampler to the SES

CAUTION: HIGH PRESSURE HAZARD

Before attaching them to the sampler, make sure the piercing valves A and M are in the full open position. If they are closed, the sampler may be depressurized unintentionally while the valves are being attached to it. This is dangerous! Also open needle valve 1.

Take the sinker bars off the sampler. Remove the retaining screws at sampler junction H-H (Fig. 2), and remove the upper part of the sampler. Remove the retaining screws at sampler junction A-A, and remove the bullnose. Fasten the sampler to the sampler mounting bracket on the backside of the sample extraction system support plate. Initially, the movable end of the sampler should be in the down position, resting on the fixed lower support bracket; i.e., the end of the sampler near the gas cyclone should point up. Remove the valve plugs (18B3522) from either end of the sampler. *After verifying that both piercing valves are fully open*, screw them on to the sampler at either end. Using the Teflon-lined, braided steel hose provided for this purpose, connect needle valve 1 to the bulkhead union fitting on the backside of the support plate. Tighten the Swagelok and Gyrolok fittings with a wrench.

Removing air from the SES

Proceed to flush the sub-boiling evaporator (H) and one or more modified gas washing bottles (I) with helium. Close valves 2, 7, 11, 15 and 21. Remove the hose clamps on the Tygon tubes attached to the bottles and the sub-boiling evaporator. Loosen the female Cajon nut that blocks off the side arm of the sub-boiling evaporator. (Gas must be able to flow out of there.) Open the regulator flow control valve, and SES valves 4, 6, 14, 8 and 23. Begin gas flow by

opening valve 16, a slow acting needle valve that may require several full turns before gas flow is observed. Adjust valve 16 and the regulator to give the gas flow desired. After several minutes flushing time, adjust the regulator to give a gas pressure slightly above atmospheric, as indicated by pressure gauge Q. Close valves 8 and 23, and clamp the gas exhaust tube of bottle I. Block the side arm on the sub-boiling evaporator by finger tightening the Cajon nut. Close valve 16 completely.

Proceed to evacuate the system and check it for leaks. Turn on the vacuum pump. Open all valves in the system except 8, 11, 16 and 23. The reading on pressure gauge P should drop to near zero. Usually, there will be some water within the SES, and the lowest pressure attainable will be approximately equal to the vapor pressure of liquid water at the given temperature. This will typically be about one-half psia. After the pressure reading on gauge P has stabilized, close valve 17. Observe the pressure reading for at least 10 minutes to verify the absence of air inleakage. If the pressure reading increases, find and correct the leak. Large leaks will be obvious. Small leaks may be found by using the valves in the SES to isolated various parts of it. The sound of the pump will usually indicate if leaking air is reaching the pump.

Collecting the gas sample

Record the pressure reading. Call this pressure value P_0.

After the system has been demonstrated to be leak tight and well evacuated, proceed to collect the gas sample. HANDS-OFF the piercing valves A and M until instructed! Close needle valve 1. Open valve 17. Isolate cryotraps F by closing valves 21 and 22. Close valves 2, 7 and 20. Now the gas handling system is isolated and ready for use. The liquid handling system is still connected to the vacuum pump.

Normally, only one gas bottle E will be used to collect a sample. Leave one valve 19 open and close the other valve 19.

Verify that valve 1 is closed. Slowly and carefully operate piercing

valve A to puncture the rupture disk inside the sampler. Verify that valves 2, 7, and 20 are closed. Verify that one valve 19 is open, and the other closed. Carefully open the needle valve 1, allowing gas to flow into the cyclone separator (B) and gas bottle E. Some liquid will be carried with it, and will accumulate in the separator.

After the gas pressure has equilibrated, record the pressure reading, and close valve 19. Call this pressure reading P_f. Also record the temperature of the SES.

Be absolutely certain that the sampler has been depressurized before proceeding further.

Collecting the brine samples

Raise the opposite end of the sampler and rest it on the upper bracket. Isolate the brine sample containers by closing valves 10, 9, 12 and 13, and verify that valves 8, 11 and 23 are closed. Close valves 2, 4, 5, 6, 7, 17, 18 and 20. Open valve 15. Operate piercing valve M. Open valve 18. Assist brine transfer with gas pressure by opening valve 16. Brine should begin to flow into the separator. If necessary, adjust the gas pressure at the regulator, but be careful not to exceed 15 psig. (The range of the upper pressure gauge is 0-30 psia, and a safety relief valves are preset to 22 psig.)

Complete brine transfer will take several minutes. If the opaque stainless steel separator is used, brine and gas flow may be monitored using a stethoscope. After brine transfer has been completed, close valves 1 and 18.

Filter the brine and transfer it from separator B to reservoir D by opening valves 2 and 3. If necessary, assist brine transfer to D with gas pressure by opening valves 7, 14 and 16.

If the filter appears blocked, bypass it by opening valve 5. If gas pressure inside D impedes brine transfer, remove the gas by closing valves 3, 7, 16, 18 and 20, and opening valves 6, 14, 15, and 17.

After all of the brine has been transferred to D, open valve 4. Partially

fill one or more of the liquid bulbs K by opening their valves (10). CAUTION: do not fill the bulbs completely; if completely filled, they may rupture if later warmed. Fill the stripping vessel J about one-half full of brine by carefully and briefly opening valve 9.

Measuring pH and filling containers I and H requires the assistance of gas pressure. To apply gas pressure, close valves 3, 5, 7, and 15. Open valves 4, 6, 14 and 16. Open valve 11 to flow brine past the pH electrode until the reading stabilizes. Close valve 11, and record the pH. Remove the tubing clamp from the gas exhaust tube connected to bottle I. Open valve 8 partially to fill the bottle. Close valve 8, and replace the clamp when sufficient brine has been transferred. The remaining brine is transferred to the sub-boiling evaporator in the same manner (remember to loosen the nut that blocks off the side arm). Because the sub-boiling evaporator has the largest volume, it should be the last vessel filled. Indeed, it would accomodate nearly the whole brine sample by itself. Remember to retighten the nut on the side-arm when done.

Rinsing the sampler with acid and solvent

If precipitation of calcium carbonate inside the sampler or SES is suspected, rinse the sampler and SES with dilute hydrochloric acid. This procedure is exactly like the solvent washing procedure described below, and should precede the solvent wash. This acid rinse is collected in one of the small glass bulbs K for analysis.

If a liquid hydrocarbon phase is present or suspected, the inside of the sampler and SES should be washed with an organic solvent to remove and collect the oil. Either hexane or 1,1,1-trichloroethane may be used. Hexane has the disadvantage of being flammable, while trichloroethane may attack the Viton gaskets and O-rings to some extent. The washing solvent should be collected in one of the small glass bulbs K.

Close valves 15, 16, 17, 18 and 20, and disconnect valve 18 from the body of the SES. Block off the open Cajon gland where valve 18 was removed. Open

valves 17, 1, 6, 7, 14 and 15 to evacuate the SES and the sampler. After the pressure drops below 1 psia, close valves 1, 2, 4, 6, 7 and 20. Pivot the free end of the sampler so it rests on the lower bracket again. Connect valve 18 to a Cajon gland that terminates in a glass or metal tube. Dip the tube connected to valve 18 into a container that contains the amount of solvent needed (200 mls should suffice). Open valve 18. The vacuum inside the sampler should easily pull the solvent into it. After the solvent has been sucked up, allow air to follow until the pressure inside the sampler approaches atmospheric. Close valve 18.

Slosh the solvent inside the sampler by pivoting it up and down several times. Raise the sampler to the upper bracket. Quickly open valve 1 (quick opening will better rinse the inside of the separator). The solvent should flow into the separator. Open valves 2 and 3 to transfer the solvent to reservoir D. If possible, the solvent should be directed through the filter in order to wash away any oil accumulated there. If necessary, assist solvent flow through the filter with gas pressure. Briefly open valve 5 to wash out the bypass line. Open the desired valve 10, and open valve 4 to transfer the solvent to a sample bulb. Close valve 10.

If the sampler and SES have been rinsed with solvent, the solvent should be removed immediately following this operation. Open valves 1, 2, 3, 4, 5, 6, 7, 14, 15 and 20. Open valve 17 to evacuate the system. Leave the system under vacuum for ten minutes or more to evaporate the solvent away.

Stripping gas from a sample of brine

Proceed to strip and collect the gases from the brine in stripping vessel J. Normally, only one of the two cryotraps will be used to collect a sample.

First, remove the small amount of water from the glass T above the stripper vessel J. Leave valves 13, 21 and 22 closed, and close valves 4, 6, 7 and 15. Open one or more manifold ports by removing valves 8 or small glass bulbs K. Open valve 16 and adjust the gas pressure to slightly above atmospheric. Open valves 9, 12 and 14. After all the water has been expelled, close valves 9, 14, and

16, in that order. Using a hypodermic syringe, inject a small, known amount of hydrochloric acid through the septum into J. Enough acid should be used to overwhelm buffering by bicarbonate and organic acid anions, and reduce the pH to 3 or lower.

Cool one cryotrap F using a Dewar of liquid nitrogen. After several minutes of cooling, open valves 21 and 22, on the cooled cryotrap only. Verify that valves 14 and 15 are closed. Open valves 12, 17 and 13. Slowly open valve 16. Adjust valve 16 to give a moderate gas flow rate, as indicated by bubbles inside J. (Be careful; it is easy to provide too much gas, causing the brine to be propelled up and out of J.) After a few minutes, close valves 21, 22, 13, 12 and 16. The procedure has now been completed, and you may proceed with the water rinsing procedure.

Final water rinse

Finally, remove the sample containers, and rinse the sampler and SES once or twice with about a quart of clean water. This is particularly important if an acid rinse has been performed. The system may be rinsed as far as the lower reservoir (D) by using the procedure described for washing with the solvent. When the rinse water is in the lower reservoir, close valves 3 and 6, open valve 4, and disconnect the braided steel hose from the glass T above reservoir D. This will allow the rinse water to drain out of D through the manifold G under atmospheric pressure. After removing the water, dry the sampler and SES with vacuum.

After disassembling the SES, determine the amount of brine collected in each of the various containers by weighing them.

3.6. Determination of gas to brine ratio

Let

P_0 = initial pressure, when the SES was evacuated.

Let

P_f = final gas pressure, after the sampler is depressurized.

If P_0 is near 0 psia, indicating no liquid water was present inside the SES to start with, estimate the water vapor pressure at the temperature of the SES, and increase P_0 by this amount.

Calculate the corrected pressure rise:

$$\Delta P = 0.98 \times (P_f - P_0)$$

The factor 0.98 corrects for the known inaccuracy of the pressure gauge.

The total gas volume in the SES is the sum of the separator volume plus the gas bottle volume:

$$V_{ses} = 2.232 + n_b \times 1.848$$

where n_b is the number of gas bottles used to collect the sample (either 1 or 2). Fortuitously, the two separators (glass and stainless steel) have the same volume.

Convert the amount of gas collected to liters of dry gas at STP using the formula

$$V_{stp} = V_{ses} \frac{\Delta P}{14.7} \times \frac{492}{T(F^\circ) + 460}$$

The sample volume of the sampler is approximately 0.98 liters. To calculate the ratio of STP gas volume to downhole brine volume, divide V_{stp} by 0.98. To convert this ratio to SCF/bbl, multiply it by 5.614.

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FIGURE CAPTIONS

Figure 1. Full scale cross-sectional drawing of the sampler that accompanies this document.

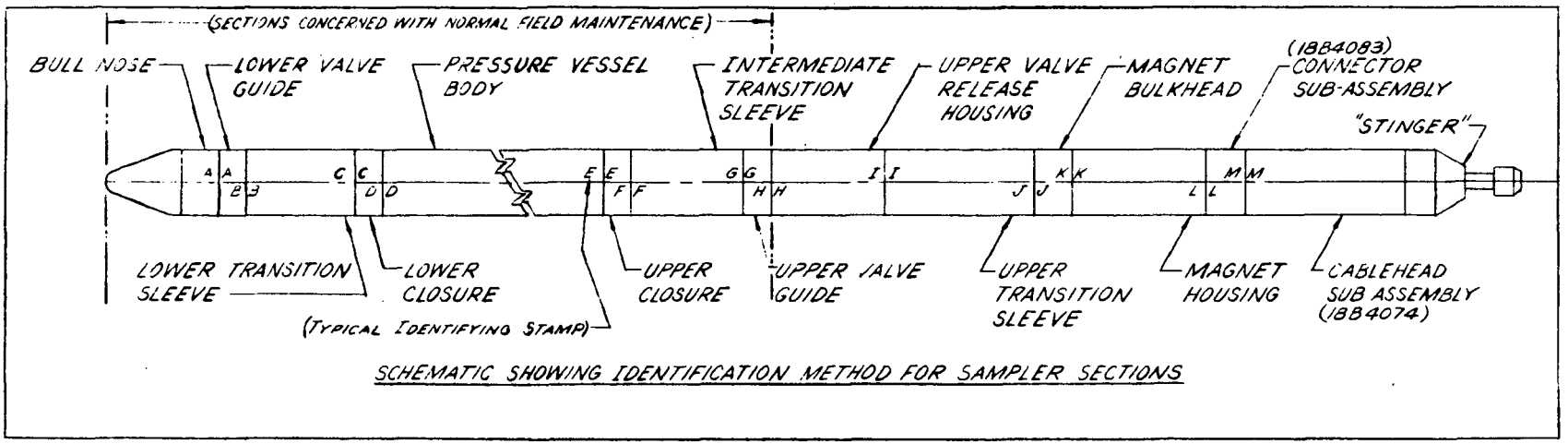
Figure 2. Simplified schematic of sampler showing how sampler sections are identified.

Figure 3. The sample extraction system in its minimal configuration. Black dots represent glass valves. Open dots represent stainless steel bellows valves. Small crossed open dots represent needle valves. All three connections to the cyclone separator (B) are armored Teflon hoses.

1 = needle valve that controls depressurization of the sampler (MP35N).
A = upper piercing valve (MP35N).
B = cyclone separator (glass or stainless steel).
D = lower brine reservoir (glass).
E = gas sample bottles (stainless steel).
G = brine manifold (stainless steel).
H = sub-boiling evaporator (glass).
I = modified gas washing bottle (glass).
K = small glass bulbs.
L = flow-through block for pH electrode (Lucite).
M = lower piercing valve (MP35N and stainless steel).
P = precise pressure gauge (0 to 30 psia).
Q = small pressure gauge (-15 to 30 psig).
R = pressure relief valve (preset to 22 psig)

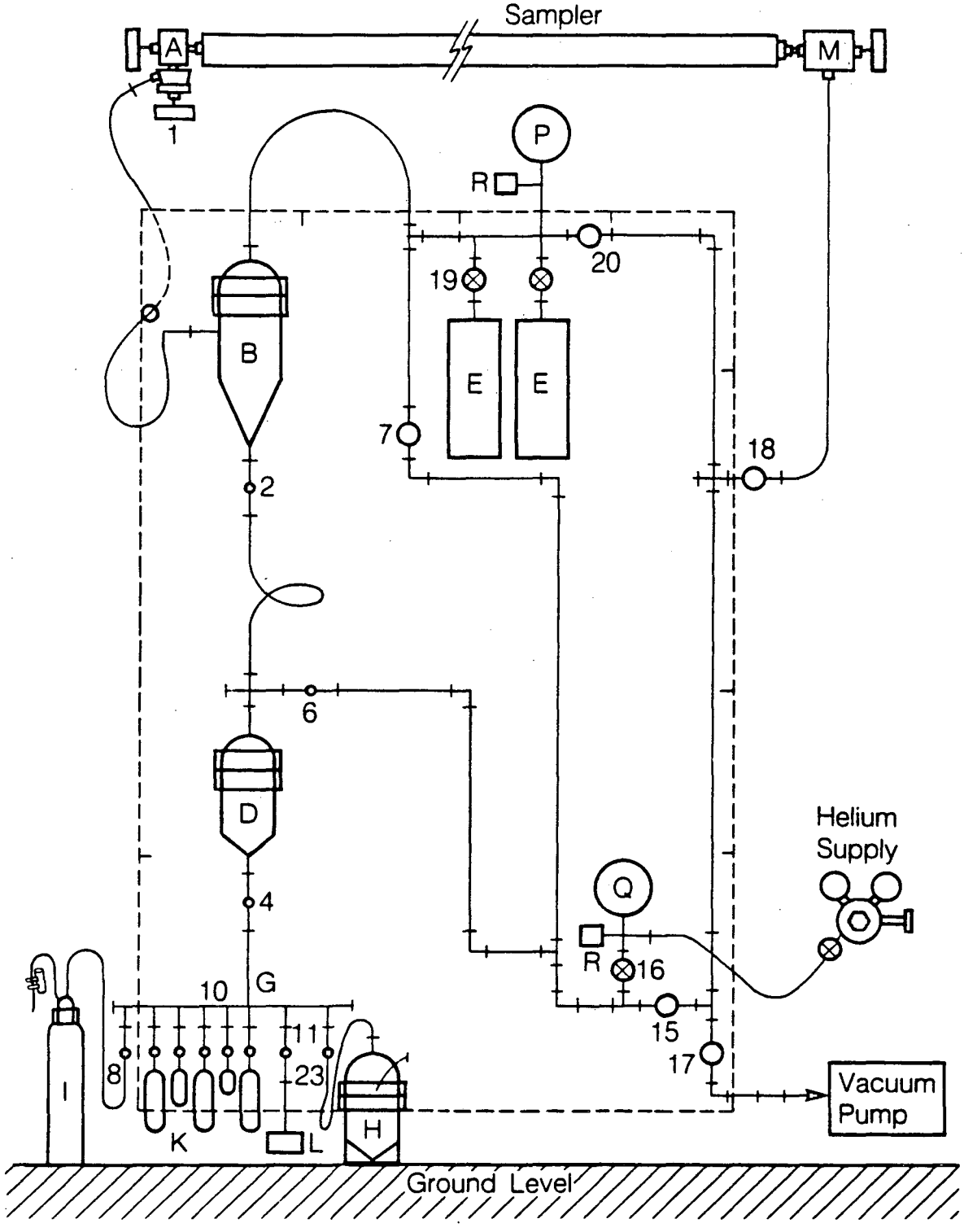
Figure 4. The sample extraction system in its maximal configuration. The filter bypass is an armored Teflon lined hose.

C = filter housing (stainless steel).
F = cryotraps (stainless steel with copper gaskets).
J = gas stripping vessel (glass).



SCHEMATIC SHOWING IDENTIFICATION METHOD FOR SAMPLER SECTIONS

Fig. 2



XBL 846-8964

Fig. 3

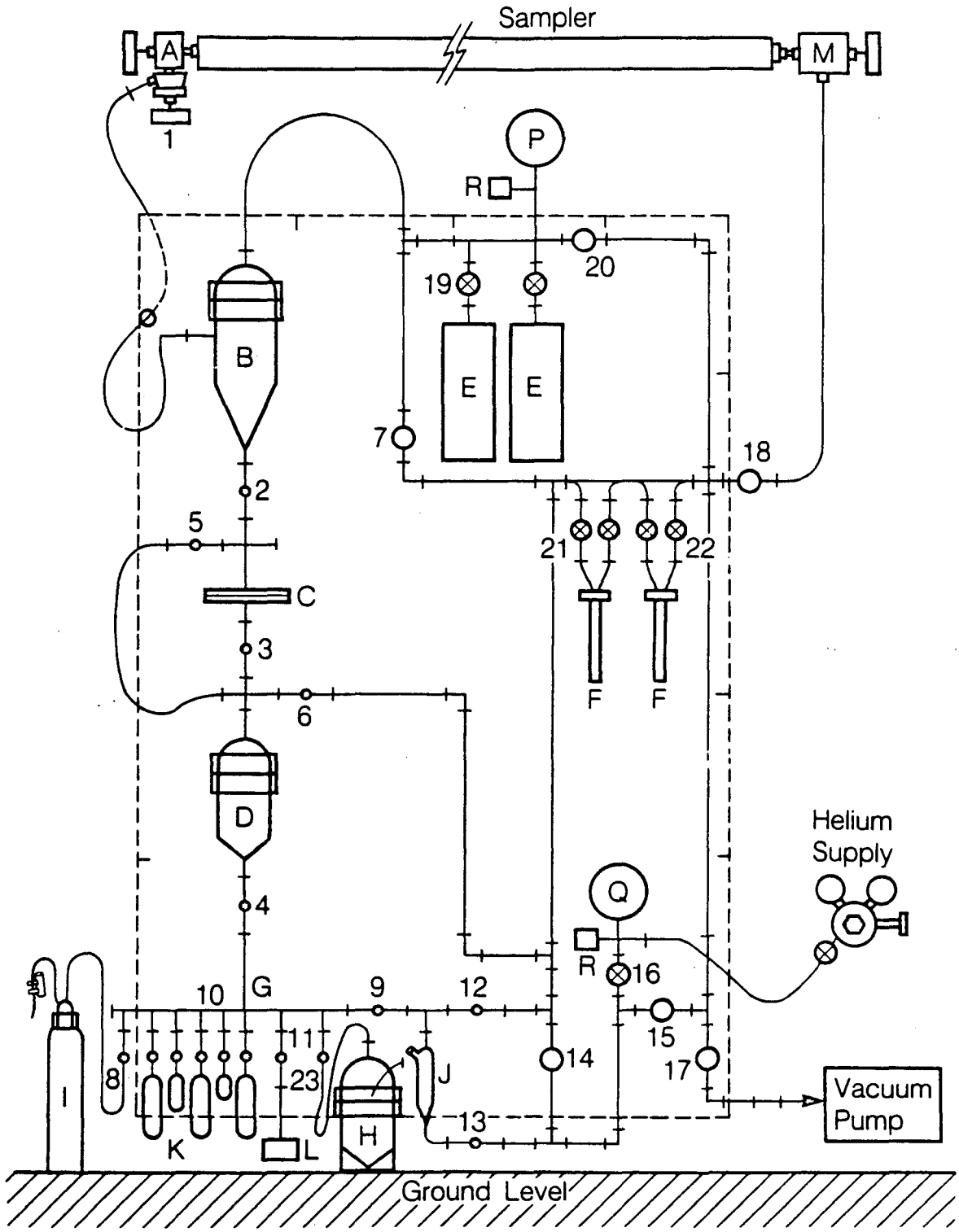


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Fig. 4

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