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A Well-Logging Probe for Measuring Tritium

Construction and Operating Manual

C. Menninga
R. L. Brodzinski

April 1983

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
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Pacific Northwest Laboratory
Richland, Washington 99352

To be used in conjunction with the
companion design manual, PNL-4069

TABLE OF CONTENTS

I. SUMMARY

This document describes the as-built construction and operating procedures for a well-logging instrument capable of measuring tritium in situ in a well or bore hole as small as 3-inch schedule 40 pipe. A companion document, "A Design Manual for a Well-Logging Probe Capable of Measuring Tritium," PNL-4069, should be referred to for all design information and drawings. This document contains sections describing changes made between the design and construction phases, the general configuration of the instrument, and step-by-step operating procedures.

The instrument can sample air or water and can purify the sample from other radionuclides or chemical contaminants. The instrument will operate satisfactorily in the presence of a moderate gamma-ray background and can measure tritium concentrations as low as 50 pCi/ml of water in normal logging operations.

II. DESIGN NOTES

The final design for the well-logging probe capable of measuring tritium is only slightly modified from that proposed in PNL-4069 (October 1981).

Modifications include the following:

A. The cold trap was redesigned, and now is a rectangular copper tank with an internal volume of 33 cm^3 (Figure 1). The gas inlet consists of 1/8" diameter stainless steel tubing wrapped with Teflon tape for thermal insulation inside 1/4" diameter copper tubing 2-1/2" long. The copper tubing is soldered into the cold trap inlet. This design has successfully avoided blocking of the air inlet by frost.

B. Of the available alternatives, the reduction step was selected in which the sample water from the cold trap is passed over hot magnesium and converted to hydrogen gas. Each charging of the furnace will process 10-12 samples.

Reduction of the water sample by passing over calcium hydride is probably feasible but has not been tested.

C. A tank of silica gel was inserted between the reducing furnace and the proportional counter to ensure that no water would get into the counter.

D. The optimum gas mixture for proportional counting of tritium in this instrument consists of 300 mm pressure of hydrogen and 450 mm of P-10 counting gas (90% argon, 10% methane).

III. CONSTRUCTION

The schematic arrangement of the probe components, connections, and valves is shown in Figure 2. Several features are worthy of note.

The flow of gases through the system is accomplished by pressure differences and electrically operated solenoid valves. A vacuum hose connecting the instrument to a vacuum pump at the surface allows various sections of the instrument to be evacuated by operation of the appropriate solenoid valves. Vacuums readily achievable with a roughing pump are adequate for all operational steps.

A pressure indicator with remote readout at the surface is connected at three different points so that the pressure can be measured for various sections of the instrument. Some parts of the operating procedures make use of these pressure measurements.

The interior of the canister can be evacuated, and the canister can be filled with nitrogen gas during operation as a safety precaution. When the probe is submerged under water, the internal pressure may be increased as a precaution against water leakage into the canister.

The hydrogen metering valve is set to deliver 4 ml of hydrogen gas per minute from a supply line at 5 psig into the air inlet line which is at 1 atmosphere. Carrier hydrogen is added during air sampling to ensure conversion of all elemental tritium to water vapor by passage over a palladium catalyst.

The sample metering valve is set to deliver 2 liters of gas per minute into the cold trap from the air sample line or nitrogen supply at 1 atmosphere. With the vacuum pump at the surface, the pressure drop across the sample metering valve is from 760 mm Hg to about 200 mm Hg.

The physical layout of components is shown in Figure 3. With the probe in the vertical operating position in a well, the sample is taken at the lower end of the probe. The tubing ports from the samplers are sealed with Swagelok fittings and Teflon ferrules.

The reducing furnace is placed at the lower end of the canister so that no electrical leads pass through that portion of the probe. The cold trap is placed as close as possible to the reducing furnace. Tubing and valves from valve #11 to the reducing furnace, except for the cold trap, are wrapped with heating tape. The silica gel tank is placed as close as possible to the cold trap, and the proportional counter as close as possible to the silica gel tank. All other components are arranged in a compact arrangement above the proportional counter.

The canister is made from 2-1/2-inch Schedule 10 stainless steel pipe. It is fabricated in three sections which are connected by means of collars with O-ring seals. Removing the sampler guard and the lower canister section provides access to the reducing furnace. When needed, the next section can be removed for access to the tank of silica gel.

The assembled probe is 3.91 m long. The "main line" components are shown in Figure 4, and the complete assembly prior to insertion into the canister is shown in Figure 5. All parts not specifically identified in this document or in PNL-4069 are generic in nature and may be replaced with any similar component. Hence, a detailed parts list is not furnished.

IV. OPERATING PROCEDURES

Caution: Before lowering the instrument into a well less than 4-inches in diameter, lower a dummy probe of the same dimensions into the well to make sure that there is adequate clearance.

Note: Refer to Figure 2 in reviewing and using these Operating Procedures.

A. Preparation for Sample Collection

1. Connect electrical supply. Make certain all valves are closed. Turn on pressure indicator.
2. Connect hydrogen line to hydrogen source. Hydrogen source supply pressure should be 5 psig.
3. Connect P-10 line to P-10 source. P-10 source supply pressure should be 5 psig.
4. Connect nitrogen line to nitrogen source. Nitrogen source supply pressure should be 5 psig.
5. Connect vacuum line to vacuum source.
6. Open valves 1, 2, 7, 6, and 5 and evacuate.
7. Close valves 1 and 7.
8. Open hydrogen supply valve to fill line.
9. Close hydrogen supply valve.
10. Repeat steps 6 through 9 two times.
11. Close valves 2, 6, and 5.
12. Open hydrogen supply valve.
13. Open valves 1 and 3 and evacuate.
14. Close valve 1 and open P-10 supply valve.
15. Close valve 3.
16. Open valves 21, 9 and 10 and evacuate.

17. Close valve 10.

18. Open nitrogen supply valve.

19. Close valves 9 and 21.

20. Open valve 19 and evacuate.

21. Close valve 19.

22. Open valve 16 and allow pressure to equilibrate at 5 psig.

23. Close valve 16.

Note:

a. During operation equilibrate N_2 pressure occasionally by opening valve 19 for a few seconds and repeating steps 21-23.

b. Maintain positive canister pressure which is dependent on depth when sampling under water.

c. Changes in canister pressure as a function of depth can be monitored with the Pressure Indicator by keeping a constant reference pressure in the sample line (valves 17, 18, and 20 closed).

24. Open valves 1 and 17 and evacuate.

25. Set zero adjust on the pressure indicator readout instrument.

Note:

a. This setting must be rechecked frequently during operation.

b. The Pressure Indicator must be calibrated according to the manufacturer's instructions. This should be done under normal operating conditions, and recalibration may be needed if

the internal canister pressure has changed appreciably due to operation under water.

26. Open cooling water valve to cold trap.

    Caution: Never apply current to the thermoelectric devices without cooling water flowing.

27. Open valves 22, 4, 13, 12, 11, and 8 and evacuate.

28. Close valve 1.

29. Open valve 2.

30. Close valve 2.

31. Open valve 1 and evacuate.

32. Repeat steps 28-30.

33. Close valves 22, 4, 12, 11, 8, and 17.

34. Open valve 14 and evacuate.

35. Set reducing furnace temperature controller to 550° C.

36. When furnace temperature reaches 550° C gradually increase to the operating temperature of 590° C.

37. Adjust heating tape to 120° C by controlling with the Variac.

    Note: Temperature can be monitored using the remote readout thermometer.

38. Adjust cold trap temperature controller to the lowest temperature reached by the cold trap.

    Caution: Make certain cooling water is flowing.

B. Air Sample Collection

1. Close valve 13.

2. Open valves 7 and 15 for a few seconds to purge the entrance lines and then close.

3. Open valves 10, 12, 11, 8, and 18 and evacuate.

4. Close valve 10.

5. Open valves 5, 6, and 15.

Note:

a. The normal sample collecting time is 25 minutes. The sample metering valve is set to deliver 2 liters per minute of air.

b. Monitor the pressure during collecting, alternating between opening valve 18 and valve 20. A drop in pressure at valve 18 indicates that the air inlet is blocked, probably by the closing of the float valve which will close the air inlet if the sampler has reached liquid water. A drop in pressure at valve 20 with no decrease at valve 18 indicates some blockage at the cold trap. Should frost block the cold trap inlet, relieve the blockage by warming the cold trap to a few degrees above 0° C with air flowing, then cool the trap and continue collecting sample.

c. While the air is being drawn into the sample inlet, the instrument should be lowered down the well at a rate that displaces approximately 2 liters per minute of air, as follows:

Well Diameter (inches)	Rate (cm/min)
3	50
4	25
≥6	10

6. Close valves 15, 6, 5, and 18 to terminate sample collection.
7. Open valve 20 and evacuate.
8. Close valves 8, 11, 12, and 14.
9. Open valves 1 and 17 and evacuate.
10. Close valve 1.
11. Open valves 2, 20, and 12.
12. Close valve 20.
13. Open valve 14 and evacuate.
14. Close valve 14.
15. Open valve 20.
16. Close valves 2, 17, 20, and 12.
17. Go to Section D.

C. Water Sample Collection

1. Open valves 20, 14, 12, and 11 and evacuate.
2. Close valves 11 and 14.
3. Open valves 10, 9, and 21 and purge with nitrogen for 5 minutes.
4. Close valve 10.
5. Open valves 11 and 14 and collect sample for 25 minutes.
6. Close valves 9 and 21 and evacuate.
7. Close valves 11 and 14.
8. Open valves 2 and 17.
9. Close valves 12, 20, 17, and 2.

D. Sample Reduction and Transfer into Counter

1. Open valves 14 and 13 and evacuate.
2. Open valves 17, 22, and 1 and evacuate.

3. Open valve 12 and evacuate.

4. Close valves 1 and 14.

   Note: Check that furnace temperature is at 590° C and that heating tape temperature is at 120° C.

5. Open valve 4.

6. Warm the cold trap to evaporate the sample. Monitor the pressure and cold trap temperature until the pressure reaches 300 mm Hg or the cold trap temperature reaches 110° C.

7. When either of the conditions under 6 is reached close the valves 4, 22, 13, and 12 in that order and discontinue heating the cold trap.

   Note: Record pressure reading if less than 300 mm Hg and add H_2 gas by opening valve 22 and intermittently opening valve 2 until the pressure reaches 300 mm Hg. Then close valves 22 and 2.

8. Open valve 1 and evacuate.

9. Close valve 1.

10. Open valve 3 to fill line with P-10.

11. Close valve 3.

12. Open valve 22 and intermittently open and close valve 3 until the pressure reaches 750 mm Hg.

13. Close valves 3, 17, and 22.

14. Open valves 14 and 13 and evacuate.

15. Open valves 12, 11, 9, and 21 and purge with nitrogen for 2 minutes.

16. Close valves 14, 13, 12, 11, 9, and 21.

E. Sample Counting

1. Set high voltage and amplifier gain settings to the indicated values.
2. With the timer preset to 25 minutes, turn on the scaler/counter.

   Note: While a sample is being counted, the next sample may be collected.
3. When the count is finished, record the total number of counts along with all other pertinent data regarding the sample.
4. Turn off the high voltage.
5. Open valves 1, 17, and 22 and evacuate.
6. Close valve 1.
7. Open valve 2.
8. Close valve 2.
9. Open valve 1 and evacuate.
10. Close valves 1, 17, and 22.

F. Background Counting

Note: The background should be determined under instrument operating conditions and, therefore, should be measured at some convenient time during the actual well-logging operation.

1. Open valves 1, 17, and 22 and evacuate.
2. Close valve 1.
3. Intermittently open and close valve 2 until the pressure reaches 300 mm Hg then close valve 2.
4. Intermittently open and close valve 3 until the pressure reaches 750 mm Hg then close valve 3.

5. Close valve 22.

6. Count background as in sample counting procedure above.

APPENDIX I

RECHARGING THE REDUCING FURNACE

The reducing furnace must be removed after processing 10-12 samples in order to recharge it with magnesium chips. After the furnace is thoroughly cooled, remove the sampler guard and the sampling apparatus. Loosen the sampler lines, which are sealed into the end of the canister with Teflon ferrules in drilled-out Swagelok fittings. Remove the end piece of the canister to gain access to the reducing furnace.

Clean the reducing furnace, and recharge it with a mixture of about 75% Mg chips and 25% pumice or lava chips. Plug both ends of the furnace with stainless steel wool. Put a little powdered graphite on the threads and contact surfaces of the fittings during reassembly of the furnace.

It is convenient to have 2 or 3 replacement furnaces already charged so that a spent furnace can be quickly replaced and can be recharged at a more convenient time.

APPENDIX II

PERFORMANCE TESTS

A prototype instrument has been constructed, and its performance has been tested. Water samples having known concentrations of tritium were introduced into the cold trap and were processed under normal operating conditions.

The sensitivity of the instrument is about 50 picocuries per milliliter of water sample, and the count rate of the proportional counter has been shown to be linear over the range 50-800,000 pCi/ml.

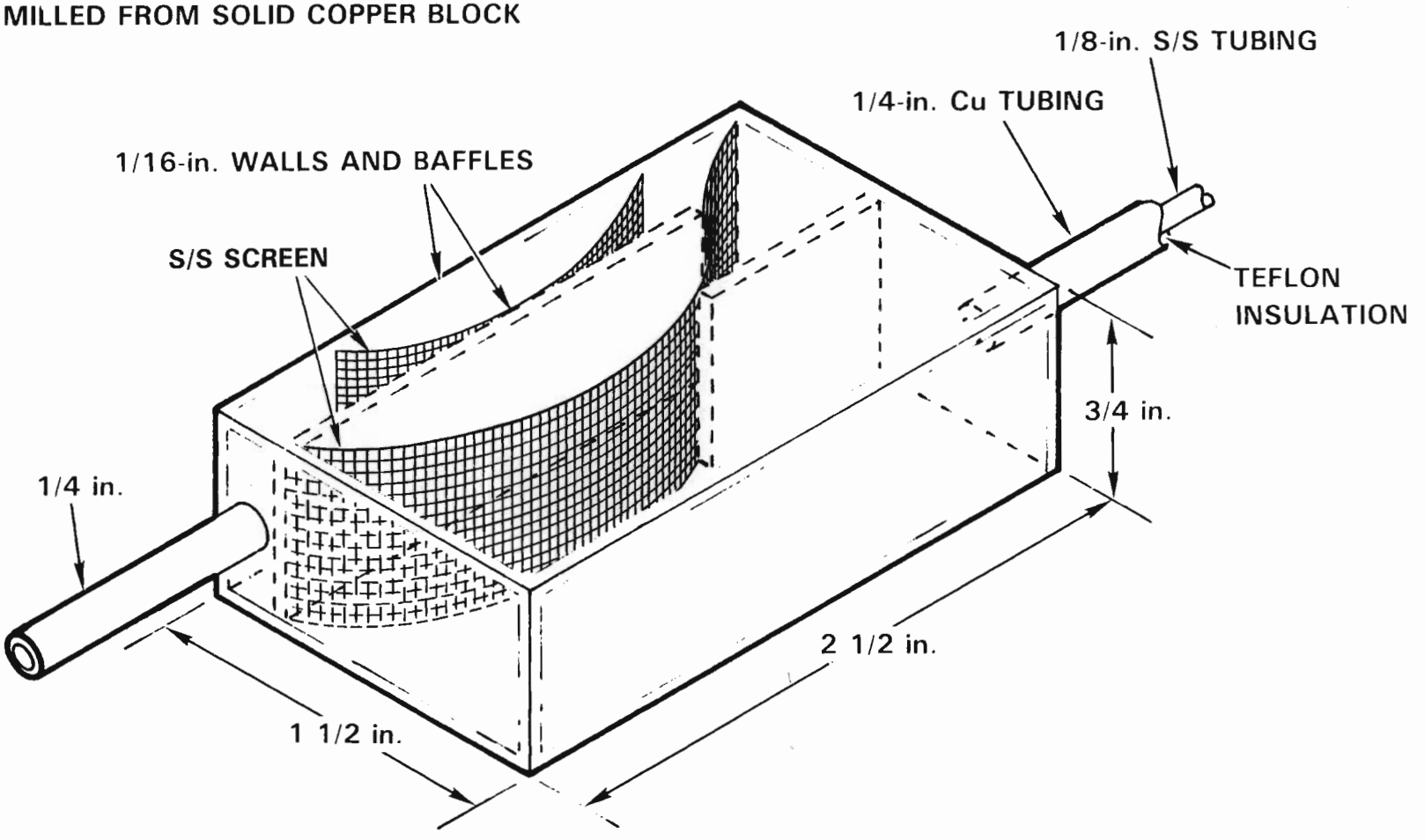

FIGURE 1. Final cold trap design

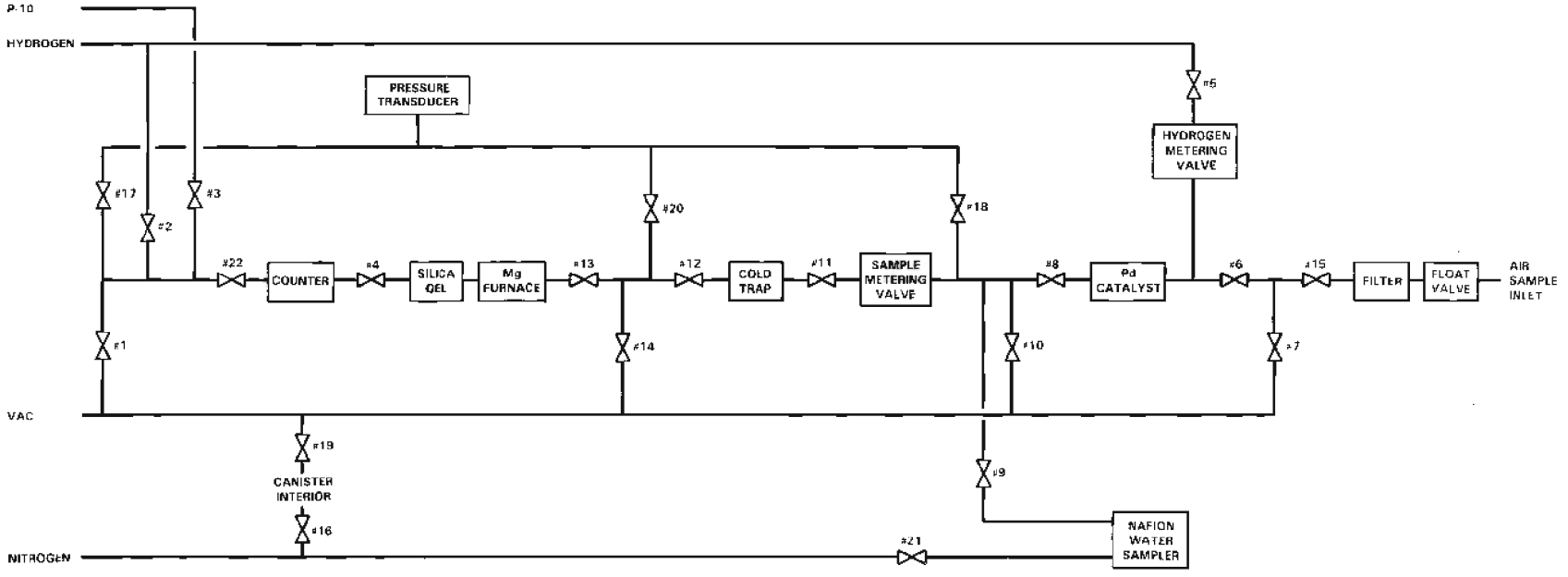

FIGURE 2. Schematic arrangement of probe components, connections, and valves

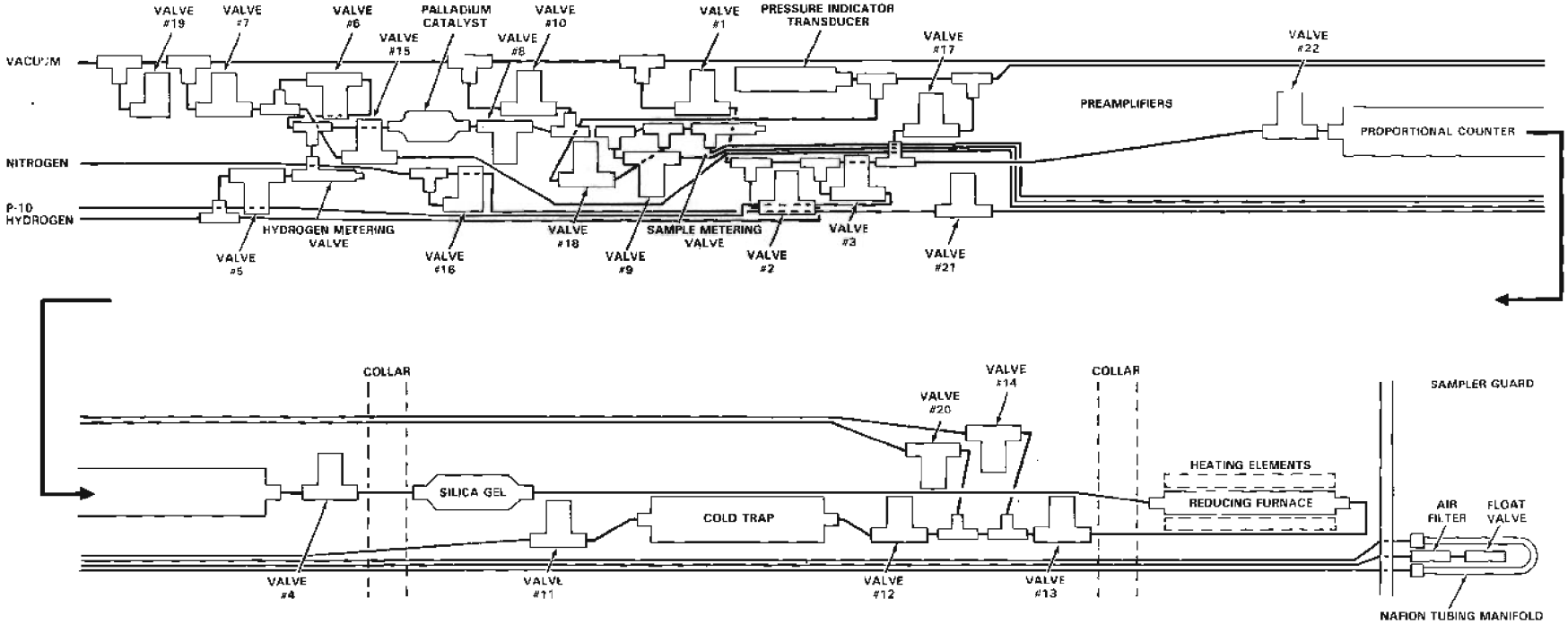

FIGURE 3. Physical layout of the components

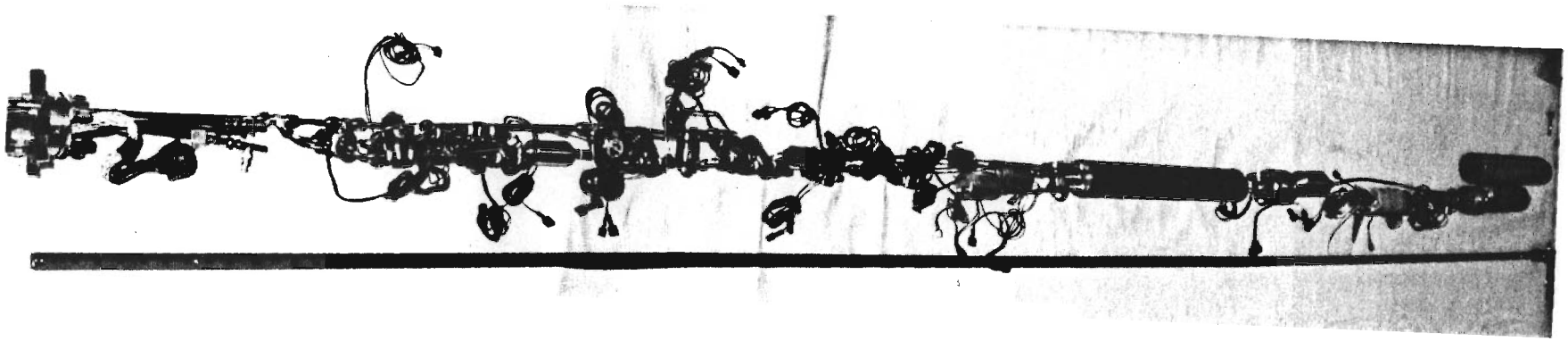

FIGURE 4. Photograph of "main line" components assembled

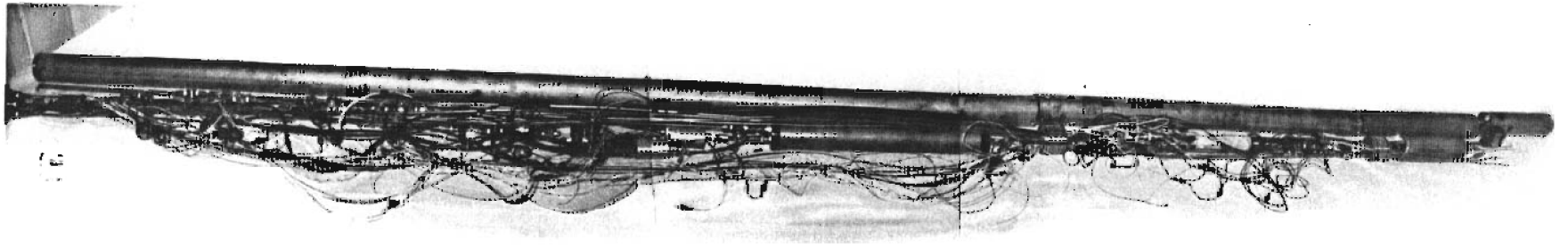

FIGURE 5. Photograph of the complete assembly prior to insertion in the canister body which is also shown

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