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7. Abstract <p>A Retained Gas Sampler (RGS) System is being developed to extract and analyze gas retained in waste tank core samples. A prototype of the separator section of the RGS system was constructed and tested.</p> <p>The results of those tests show that 99.7% of simulated retained gas could be separated from the extractor portion of the RGS system in only 10 cycles of a liquid mercury positive displacement pump.</p> <p>The tests also produced valuable information regarding selection of materials and components.</p> <p>The testing and operation of the prototype separator section also provided insight for the authentic system's operating procedures.</p>		
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MASTER

1.0 SUMMARY

A test system was constructed which simulated the separator section of the proposed Retained Gas Sampler (RGS) system for proof-of-principle testing. The laboratory setup was designed to duplicate the physical conditions of the RGS separator section in dimension, construction material, and techniques of operation.

The test results demonstrated that a 40 mL aliquot of gas at atmospheric pressure (approx. 1.6 mmoles), placed in an evacuated chamber resembling the extractor section of the proposed RGS system which was filled with 300 mL water, could be pumped from the extractor to a collector with 99.7% recovery in 10 cycles using a mercury displacement pump.

In general the test results showed that the method of mass transfer and specifically the design and operation of a mercury positive displacement pump could quantitatively transfer small or large amounts of gas, under vacuum and in a high water vapor pressure environment, to a collection vessel suitable for transport to offsite analytical instrumentation or immediate (online) analysis.

The effectiveness of the RGS system will depend greatly upon the ability to construct a system which is capable of maintaining tight vacuum integrity. The use of Cajon® type tube connectors where fittings would experience torque, flexion, and repeated disconnection and reconnection, the minimal use of plastic tubing, and the complete exclusion of vapor from the headspace of the mercury pump will contribute greatly to high quality system performance.

2.0 INTRODUCTION

Radioactive waste materials in underground high level waste (HLW) storage tanks at the Hanford Site evolve gaseous mixtures at varying rates. In order to verify the flammability of these gases and the mechanisms by which they are produced, it is necessary to sample material from these tanks in such a way as to preserve the gas phase of the material for analysis. Careful laboratory studies could then be performed on these samples which would allow judgement to be made of the hazard level of the storage tank.

The Retained Gas Sampler (RGS) system is such a sampling method. A multi-disciplinary team developed and issued a plan (Reference 1) to obtain waste tank core samples for gas phase analysis. This plan contained the basic idea and function of the RGS system. Different organizations assumed responsibility of various aspects of the RGS program which they were most qualified to develop.

In order to determine the feasibility of extracting, isolating, and analyzing gas from a waste tank sample, a method for performing these functions needed to be devised, tested, and developed. Two ideas emerged which supported this end.

One idea involved an out-of-hot cell method which employed a "Van Slyke Apparatus." This device required a 10 mL sample of waste tank material to be "placed" inside the apparatus. Through careful manipulation and adjustment, the apparatus would separate the retained gas from the bulk of the sample which could then be analyzed. There were two drawbacks to this method: 1) it required extensive hands-on operation (in a hood) and, depending upon the sample, could increase substantially the hand dose of the operator, 2) the Van Slyke Apparatus could not determine the amount of free or unbounded gas which might accompany the sample.

The other method considered was one which employed an extractor device (inside the hot cell) which would receive the waste tank sample remotely and, through remote manipulation, would separate the gas from the bulk of the sample. By using a positive displacement pump, the gas would be transferred to a collection vessel (outside the hot cell) where it would be quantified and analyzed. The extractor device would be able to differentiate the various types of gases associated with the sample, i.e. it would independently separate from the bulk sample the free or unbound gas, the physically bound gas, and the chemically bound gas components--all of which could be independently analyzed.

The decision was made to pursue the latter of these two methods for gas extraction. Among other things, development of a mercury positive displacement pump and its operation commenced right away. A test plan (Reference 2) was written to test the proof-of-principle of using a liquid mercury positive displacement pump for mass transfer and to test other aspects of the separation and isolation process. This report contains the results obtained from that test plan.

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Though not all of the tests outlined in the test plan were performed, enough research was conducted to determine whether or not this type of mass transfer operation should be attempted in an authentic application.

3.0 SCOPE

The development of the RGS extraction device was divided into three phases. The table below lists each phase and identifies (•) the principle objectives of each phase.

Table 3.1 Laboratory Development Scope

Phase I: GAS PHASE MASS TRANSPORT EVALUATION <ul style="list-style-type: none">• Operating Parameters Test PlanLaboratory System SetupLaboratory Testing★ Test Results Report
Phase II: FULL SCALE PROTOTYPE EVALUATION <ul style="list-style-type: none">• Waste Tank Simulant Test PlanFull Scale RGS System SetupFull Scale RGS System Testing• Test Results Report
Phase III: AUTHENTIC APPLICATION EVALUATION <ul style="list-style-type: none">• Authentic Application Test PlanSample AcquisitionPerform Separation and Analysis• Separation and Analysis Report

(★) This document reports the results of work conducted under the Phase I Test Plan.

4.0 OBJECTIVE

There were two objectives for the experiments conducted in this document. One objective was to develop a method which would determine the amount (moles) of gas in a given waste tank sample. The other objective was to develop a method which would transport these gases from the bulk sample to a collection vessel or analytical instrument. Successfully accomplishing these goals would indicate that qualitative and quantitative analysis of the retained gas via these means was possible and practicable.

Several ideas for accomplishing these goals had already been formulated but not yet proven to work. The test plan (Reference 2) was written to effect the proof-of-principle of these ideas and this document reports the results of those tests.

A series of experiments were conducted on a laboratory setup of the separator section to determine the feasibility of this sort of mass transfer and develop a method which would efficiently carry out the mass transfer operation. Each of the experiments had a specific objective:

- Establish a data logging method.
- Determine the equipment configuration.
- Develop a procedure for pressure transducer/indicator calibration.
- Develop a procedure for the determination of the system's internal volume in sections and whole.
- Develop a system leak rate determination procedure.
- Develop and validate a procedure for mass transfer.
- Determine the mercury positive displacement pump efficiency.

5.0 EXPERIMENTAL

5.1 RGS System Laboratory Configuration

The laboratory setup was constructed as illustrated in Figure 1. This arrangement was not the only configuration explored but it proved to be the best configuration designed. Vacuum integrity was the most difficult aspect to achieve. Using parts and fittings which involved the fewest tube connections appeared to be the best strategy in battling system leaks. Complete vacuum integrity was never fully realized in this setup but leakage was (finally) reduced to workable rates.

Vacuum integrity will be of paramount concern when constructing (and using) the authentic setup. This will become more apparent during the cold testing of the full scale setup in Phase II.

Figure 5.1 Laboratory Setup Configuration

Though not shown in Figure 5.1, several types of valves were used in the laboratory setup. Valves which straddled vessels, i.e. A, B, C, G and H were bellows valves; valve F and I were 3-way valves; the remaining valves (D, E, J) were quarter turn diaphragm valves.

Wherever possible, 1/4" Swagelok^{®1} type connectors were used to connect tubing to the various components. Exceptions to this were with the 1/4" NPT fittings attached to the pressure transducers. Also, all tubing in the system was composed of 1/4" O.D. 316 stainless steel. Where glass was concerned (such as the mercury pump and sight glass) glass to metal fittings were used which allowed the glass components to be connected to the system using normal Swagelok[®] tube connectors.

The filter used in the system was a Wafergard^{®2} F Mini In-line Gas Filter WGFG 01 HS1. It was rated at greater than 99.999999% retention of particles larger than 0.003 μm .

Two pressure transducers were used in this setup, one on the extractor side and the other on the collector side of the mercury pump. Though the transducers were not the most accurate measurement devices available, they did facilitate an unbroken indication of pressure from atmospheric pressure down to 250 pascal (absolute). A BASIC program (Attachment 1) was written which allowed the indicated pressures to be automatically imported and saved to a text file on a personal computer for post analysis. This feature will be exceedingly useful during the authentic application because it affords the operator the freedom to focus on more critical aspects of the operation and not be so distracted with constant data logging.

The test plan (Reference 2) called for a very thorough investigation of the system characteristics and operational technique. Unfortunately not all of the aspects were explored--mainly because of the time required in resolving some of the associated complications. One aspect not investigated which would have been extremely beneficial was the test and evaluation of the Quad 400 Gas Analyzer³, a sensitive gas chromatograph suitable for in-line analysis. The instrument, unfortunately, possessed several internal problems and had to be sent back to the manufacturer for repair. Also, standard gas samples such as mixtures of hydrogen/nitrogen/ammonia and mixtures of nitrous oxide/nitrogen were to be run through the system to determine whether gas chemistry affected quantitative mass transfer. Unfortunately, permits for use of these gases in the laboratory were not acquired in time for the report. Finally, a heater on the system was not installed due to thermostat control problems. Thus, all tests performed in these experiments involved only air as the sample gas at room temperature (~25°C).

¹Swagelok[®] is a registered trademark of Swagelok Company.

²Wafergard[®] is a registered trademark of Millipore Corporation.

³Quad 400 Gas Analyzer is a product of MTI Associates.

5.1.1 Extractor Section

The laboratory setup included a 1000 mL vessel which served to simulate the proposed gas extractor section of the RGS system. It did not contain any of the extractor's hardware such as the sampler port, mixer bearing/motor or secondary volume but it allowed aqueous solutions to be placed inside the volume so to simulate the vapor pressure characteristics of waste tank materials.

The extractor section included those components of the laboratory setup from valve A to valves E and F (Figure 5.1). All components of the extractor section were made of stainless steel and were connected together using Swagelok® tube connectors.

Connected to one end (upper end) of the vessel was a 40 mL stainless steel gas sample vessel. This was to facilitate a uniform addition of gas sample to the system. Also connected to this end of the 1000 mL vessel was a pressure transducer (PT) and an in-line filter.

The filter was installed on the tube connecting the extractor section to the separator section in order to replicate the effects that such a filter might have on the mass transfer operation of the actual setup. On the actual setup a filter will be installed on the tube exiting the hot cell which connects the extractor to the separator to prevent the release of radioactive particulates into the environment.

Connected to the other end of the 1000 mL extractor vessel was a cut-off valve with a stem extending down into a beaker for the addition of water into the vessel.

5.1.2 Separator Section

The bulk of the laboratory setup served to simulate the separator section of the RGS system. This section included the mercury positive displacement pump, a 40 mL capacity collector vessel, pressure transducer, sight glass, and mechanical vacuum pump.

The mechanical vacuum pump was used (and will be used on the actual setup) to purge air, water and other volatiles from the system for leak checks, calibration routines, and sample introduction.

The 40 mL gas collection vessel was connected in such a way that two valves straddled the 40 mL volume of the vessel and each could be closed to hermetically seal the volume of the vessel. This way, the vessel could be removed from the system setup and transported (while containing a gas sample) to off-site analytical instruments when necessary. If not removed, the vessel could collect and store extracted gas samples, or allow gas to pass completely through the system during a purge operation.

The sight glass served as a window to view the level of mercury in the separator section. Since pressure readings were the only means for quantifying the amount of gas collected, the other parameters, namely temperature and volume were required to remain essentially constant. Thus, it was important to monitor the level (volume) of mercury in the separator section. The sight glass also served as a window to determine if a sufficient amount of mercury had filled the mercury displacement pump to completely fill the volume (leaving no vapor head space).

5.1.3 Mercury Positive Displacement Pump

The mercury positive displacement pump was the device used for transferring gas sample from the extractor to the collector. Approximately 750 mL of liquid metallic mercury was contained inside a glass displacement cylinder connected to a steel reservoir via a 6 foot piece of 1/4 inch I.D., 1/8 inch wall, clear plastic, nylon reinforced tubing. The glass cylinder was fitted with a single three-way stainless steel valve manufactured specifically for high vacuum applications.

To protect against breakage and the consequential catastrophe of losing 750 mL of mercury to the laboratory floor, a thick sheath of polyester resin was cast around the glass cylinder exposing only the metal tube nipples at each end of the displacement cylinder. The volume of the displacement cylinder was about 425 mL which included the volume of the three-way valve. The other 325 mL of mercury occupied the volume of connecting tubes, the sight glass, and the reservoir itself. The three-way valve was of a design which permitted the valve to be turned through the liquid mercury thus eliminating any head space vapor in the displacement cylinder. (An inclusion of head space vapor in the pump substantially reduces the efficiency of the pump--particularly at low pressures.)

Standard laboratory bench hardware was not considered sturdy enough to safely support the completely filled displacement cylinder (or the reservoir) of mercury (750 mL Hg = 10.1 kg). Therefore a dedicated support was constructed from thick-walled steel pipe which supported both the displacement cylinder and the reservoir. The displacement cylinder was supported rigidly using various clamps which held the cylinder firmly to the pipe support itself. The reservoir had attached to it a sturdy hook which could be easily hooked to any link of a length of chain attached and suspended from the top of the pipe support. The links in the chain allowed the reservoir to be hung at various levels (higher, equal, lower) relative to the height of the displacement cylinder for pumping activities.

The accidental flooding of the system with mercury was found to be quite easily accomplished. All that was required for this to happen was for valve F to be opened to either the collector side or extractor side of the system and the reservoir placed too high on the chain (but not necessarily higher than the displacement cylinder). Unfortunately, due to the operation of the system, at least one of these conditions was necessarily and frequently assumed. To

reduce the likelihood of such a potential disaster, a general procedure was developed and, when followed, appeared to effectively reduce the likelihood of this operational and analytical mishap. The steps of the general procedure were:

1. Before the start of any experiment of operation, valve D shall be closed.
2. The mercury reservoir shall be lowered to its lowest position (>760 mL below the bottom of the displacement cylinder).
3. When the mercury reservoir is to be raised:
 - a. Valve D shall first be closed.
 - b. Valve F shall be opened toward the collector side only.
4. Near the end of the complete filling of the displacement cylinder, the rate of flow of mercury shall be reduced by lowering the reservoir somewhat. (This reduces the inertial shock when the flow of mercury is finally stopped at valve F.)
5. Once the mercury has reached its desired level, valve F shall not be turned to either the extractor side or the collector side.
6. Valve F must not be adjusted again until the reservoir has been lowered again to its lowest position.

5.2 Data Logging Method

Due to the delicate nature of the mercury pump, the pumping routine required a great deal of the operator's attention. This will be true for the actual setup as well. It was necessary, therefore, to relieve the operator of the tedious chore of data logging so that distractions from the mercury pump operation would be minimized.

The setup was therefore equipped with Omega⁴ DP-41S display panels which allowed for direct downloading of the display readout to a personal computer.

The BASIC program listed as Attachment 1 was written to perform this task. It allowed the operator to specify the rate at which the data was to be acquired and displayed the data in numerical and graphical form on the monitor. Data entered into the personal computer through com ports 1 and 2 and was saved to disk during the acquisition in a text file for post processing.

5.3 System Leak Rate

The first experiment to be conducted on the setup was one which

⁴Omega Engineering, Inc. Stamford, CT.

determined the system leak rate. This was a fairly simple exercise in which the automatic data logging software was employed to continually monitor the system internal pressure over a duration of several minutes. Because the increase in system pressure due to leakage was essentially linear with time (for slow leaks in the duration of up to an hour), the slope of the pressure/time curve could be considered proportional to leak rate ($\mu\text{moles/sec}$). A linear least squares fit of the data allowed the entire data acquisition to be consolidated to a single ratio. To determine the leak rate, a knowledge of the system volume was required. However, since individual component volumes were already known, a reasonable estimate of the system volume could be derived. Thus the leak rate L (expressed in $\mu\text{moles/sec}$) was derived from the pressure/time data acquired according to the following equation.

$$L = \frac{SV}{RT} \quad \text{Equation 5.1}$$

- Where:
- S = linear least squares slope of the pressure/time curve expressed in units of pascal/sec.
 - V = System internal volume expressed in milliliters (mL)
 - R = gas constant, $8.314 \text{ J/(K}\cdot\text{mol)}$
 - T = system temperature (Kelvin)

The leak rate determination was made by performing the following steps:

1. Close valve D.
2. Lower mercury reservoir (empty mercury from pump displacement cylinder).
3. Open all valves (except F, I)
4. Close valve A.
5. Turn valve F to disconnect pump displacement cylinder from system.
6. Turn valve I to connect system to the mechanical pump.
7. Operate mechanical pump to evacuate system down to less than 250 pascal absolute. (This took about 15 minutes.)
8. Close valve H and turn off mechanical pump.
9. Run data acquisition software and collect data.

10. Collect data for 30-60 minutes

11. Analyze data and calculate leak rate.

Variations of this experiment were conducted on specific components of the setup by isolating a section of the system with the appropriate valves. The leak rate determination procedure was then used to determine the leak rate of the isolated section.

5.4 Mercury Pump Efficiency Determination

Mercury pump efficiency was defined as the time required to pump the majority of the gas in the extractor to the collector. The size of the pump displacement cylinder needed to be large enough to evacuate the volume of the extractor in a reasonable amount of time, but not so large as to require an excessive amount of liquid mercury. Since a large pump displacement volume should effectively reduce the time involved for the mass transfer, the influence of system leaks on analytical quality would be minimized if an adequately large displacement volume were used. The efficiency determination was made by performing the following steps:

1. Close valve D.
2. Lower mercury reservoir to empty mercury from pump displacement cylinder.
3. Open all valves (except F, I).
4. Turn valve F to connect pump displacement cylinder to collector side.
5. Raise mercury reservoir to fill pump displacement cylinder to sight glass. (Control with Valve F.)
6. Close valve B, E, J.
7. Turn valve I to connect system to atmosphere.
8. Run software for data acquisition.
9. Lower mercury reservoir to empty mercury from pump displacement cylinder.
10. Turn valve F to connect pump displacement cylinder to extractor side. Wait till mercury settles to lowest level.
11. Turn valve F to connect pump displacement cylinder to collector side.
12. Raise mercury reservoir to fill pump displacement cylinder to sight

glass. (Control with valve F.)

13. Repeat steps 9 through 12 until most (>99%) of the gas is removed from the extractor.

14. Evaluate acquired data and determine pump efficiency.

5.5 Volume Determination

In order to determine the amount of bulk sample (solid/liquid phase) that is present in the extractor after extrusion, the total volume of the extractor must be known, and, after extrusion, the remaining space (vapor space) of the extractor must be determined.

Extractor volume determination was conducted by adding a known volume and known pressure of a small amount of air into the extractor volume and, through application of the ideal gas law, the volume of the extractor was calculated. The gas law equation for adding two volumes of gas at two different pressures can be represented as:

$$P_1V_1 + P_2V_2 = P_3(V_1 + V_2) \quad \text{Equation 5.2}$$

where: P_1 = pressure of the air sample prior to addition to the system

V_1 = volume of the air sample prior to addition to the system

P_2 = pressure of the air in the unknown volume prior to addition of the air sample

V_2 = extractor volume

P_3 = final pressure in the extractor volume after addition of air sample

Of course this relationship assumes that the temperature difference between the two separate volumes is negligible.

Rearranging Equation 5.2 to set V_2 as a function of all the other parameters yields:

$$V_2 = V_1 \cdot \frac{P_3 - P_1}{P_2 - P_3} \quad \text{Equation 5.3}$$

By substituting recorded values of P_2 and P_3 and known parameters P_1 and V_1

into equation 5.2, V_2 , the extractor volume was determined. Unfortunately, the type of pressure transducers that were installed on the system, though accurate enough, were not very precise and yielded values of P_2 and P_3 which exhibited too much error for a single volume determination. To get around this an averaging technique was used. This technique involved a series of air sample additions such that the final pressure (P_3) obtained after every air sample addition was substituted for the initial pressure (P_2) prior to the next addition of air sample. Running the data logging program during the experiment, a pressure/aliquot curve was generated which, when applied to Equation 5.3 yielded an average volume with only about one percent error.

The following steps were employed for the volume determination of the extractor side of the system.

1. Close valve D.
2. Lower mercury manometer to empty mercury from pump displacement cylinder.
3. Open all valves (except F, I).
4. Close valve B.
5. Turn valve F to disconnect pump displacement cylinder from either extractor side or collector side.
6. Turn valve I to connect system to mechanical pump.
7. Evacuate system to about 100 Pa (absolute) using mechanical pump.
8. Close valve D.
9. Data logging program started in "interactive" mode. (User signals input to computer.)
10. Record pressure (P_3).
11. Close valve A, open valve B, wait 15 seconds.
12. Record pressure (P_3).
13. Close valve B, open valve A, wait 15 seconds.
14. Repeat steps 11 through 13 about 20 times.
15. Analyze data and calculate volume (V_2).

5.6 Mass Transfer Simulation

The mass transfer experiment was the final experiment conducted on the laboratory setup. This experiment tested the performance of the separator section's pumping operation in a scenario which was thought to most closely resemble the conditions and procedures anticipated for the actual setup.

To prepare the experiment, the system was evacuated and then 300 mL water was added to the extractor vessel. This consequently filled the void space with water vapor which raised the pressure in the system from about 50 Pa to about 3300 Pa. An aliquot of air (40 mL, atmospheric pressure) was then added to the extractor vessel to simulate the gas phase component of the sample. This air (and water vapor) was then pumped from the extractor (using the mercury pump) to the collector. During the pumping sequence, the data logging program was run to monitor the pressure of the extractor and collector sections of the setup.

The following steps were used in the mass transfer procedure:

1. Close valve D.
2. Lower mercury reservoir to empty mercury from pump displacement cylinder.
3. Open all valves (except F, I).
4. Close valves B, J.
5. Turn valve F to connect pump displacement cylinder to collector side.
6. Evacuate system to 50 Pa (absolute) using mechanical pump.
7. Raise mercury reservoir to fill pump displacement cylinder to sight glass. (Control with valve F.)
8. Close valve H and turn off mechanical pump.
9. Fill beaker with water.
10. Open valve J and suck in 300 mL water. Close valve J.
11. Run data acquisition program and collect data in (interactive) mode.
12. Close valve A. Open valve B. Wait 15 seconds. Close Valve B.
13. Close valve E.
14. Lower mercury reservoir to empty mercury from pump displacement cylinder.

15. Turn valve F to connect pump displacement cylinder to extractor side. Wait till mercury settles to lowest level.
16. Turn valve F to connect to collector side.
17. Raise mercury reservoir to fill pump displacement cylinder to sight glass. (Control with valve F.)
18. Repeat steps 14 through 17 for about 20 times or until gas collected in collector is equal to >98% aliquot.
19. Analyzed acquired data and determine feasibility.

5.7 Pressure Transducer Calibration

Calibration of the pressure transducers was performed by using a two-point calibration method. The method used was that which was outlined in the Operator's Manual of the DP-41S display panels (Written by Omega® Engineering Inc., Chapter 8, Unipolar Potentiometric Input). The method required the pressure inputs of two different values (preferentially, those which bracketed the pressures experienced during the application). The values chosen were the current atmospheric pressure and vapor pressure of water at ambient temperature. The value of the current atmospheric pressure was obtained by calling (telephone) the site weather forecaster and asking for the current barometric reading at the 200 West area. The Second pressure was obtained by knowing the temperature of the extractor in the laboratory and reading the corresponding vapor pressure of water at that temperature as listed in the *Handbook of Chemistry and Physics*.

To perform the calibration routine, the following steps were followed:

1. Obtain current atmospheric pressure. (Convert to useful dimension).
2. Close valve D.
3. Lower mercury reservoir.
4. Open all valves (except F, I).
5. Reset DP-41S meter and record reading for atmospheric pressure.
6. Close valve B. Turn valve I to connect system to mechanical pump.
7. Turn on mechanical pump and evacuate system.
8. Fill beaker with water.
9. Open valve J to suck in 300 mL water.

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10. Determine system temperature and, using vapor pressure/temperature tables determine the vapor pressure of water. (Convert to useful dimension.)

11. Read DP-41S for evacuated system pressure.

12. With the four values obtained: actual atmospheric pressure, atmospheric pressure indication, tabulated water vapor pressure, and indicated water vapor pressure, configure the DP-41S meter according to the manufacturer's instructions.

6.0 RESULTS AND DISCUSSION

6.1 RGS System Laboratory Configuration

The best configuration obtained to simulate the extraction system was the one represented in Figure 5.1. This configuration seemed to possess vacuum leak characteristics that were most manageable and allowed the location of troublesome leaks to be determined. Unfortunately, complete vacuum tight integrity was never fully realized. Over a period of time the evacuated system would always gain in pressure due to either atmospheric leaks or internal off-gassing. Components were connected together using 1/4" outside diameter (O.D.) 316 stainless steel tubing which possessed several favorable characteristics:

1. Of the three dimensions tested, {1/4", 1/8" and 1/16"} O.D., connections made with 1/4" O.D. tubing, were the easiest and fastest to make with minimal leakage. Also, 1/4" tubing was much more "forgiving" when connections were repeatedly broken and refitted.
2. Gas transport through 1/4" O.D. tubing required considerably less time than with 1/8" or 1/16" O.D. tubing. This factor gives 1/4" O.D. tubing a distinct advantage over the other tube dimensions for minimizing the influence of system leaks on analytical quality.
3. Manifolds made from 1/4" O.D. tubing were more rigid than those made with other tubing dimensions which allowed the system to be constructed without excessive structure support.

The most reliable valve for tight vacuum integrity was found to be the Nupro⁵ "DL" series valve. These were diaphragm valves which required only a one-quarter turn to actuate the valve from fully open to fully closed. Also, the "dog-leg" handle permitted easy visual indication of the valve's open or closed configuration.

Connections made between tube and fitting (valves, unions, tees) consisted mainly of Swagelok[®] type fittings. The initial tightening of these fittings usually made connections which were satisfactory for tight vacuum integrity, but they seemed to fail about 30 percent of the time when broken and then re-tightened for whatever reason. Experiments conducted with Cajon⁶ fittings found these fittings to be much more "forgiving" when connections were repeatedly broken and remade.

Plastic tubing was used initially to connect glass components to the stainless steel tube sections of the manifold. Acceptable vacuum integrity, however, was never suitably accomplished with any sort of plastic tubing. The reason

⁵Nupro[®] is a trademark of Swagelok Company.

⁶Cajon[®] is a trademark of Swagelok Company.

for this appeared to be not so much due to the permeability of air through the tubing (which in itself was a nuisance) but more so due to leakage at the interface between the plastic tubing and stainless steel tubing. Clamps placed at the steel/glass connection points did not help because they seemed to cause "bunching" of the plastic tubing around the stainless steel tubing thus making the leak even worse.

Therefore the use of plastic tubing on the system was limited to places where no other type of tubing material would do, namely, the connecting tube between the mercury pump displacement volume and the reservoir. Elsewhere where flexible tubing was required on the system, 24" lengths of Cajon® flexible stainless steel tubes were used and presented no complications.

The best configuration of the positive displacement pump consisted of a glass cylinder (approximately 400 mL) oriented vertically and connected at the top with a stainless steel center-fed, bi-directional three-way valve (SS-43XS4). At the bottom end of the glass cylinder was connected a 6 foot length of nylon reinforced plastic tubing which connected the cylinder to the mercury reservoir. The 3-way valve on the top of the displacement volume allowed the mercury to pass cleanly through the valve so that the displacement cylinder maintained no vapor head space.

Prior to using the stainless steel 3-way valve, a 3-way glass stopcock valve was used on the displacement cylinder. The glass stopcock was attached as a solid extension of the cylinder (glass welded) with vacuum grease to seal and lubricate the stopcock in the body. The glass stopcock worked well enough and maintained tight vacuum integrity when freshly lubricated. However, vacuum grease would eventually work its way into the pump cylinder and tube and would effectively plug the flow of gas at low pressures.

The stainless steel 3-way valve presented no grease problems as the glass stopcock valve did. Also, there were no complications associated with mercury/steel amalgamation or valve seat problems that might have been caused by exposing and turning the valve through liquid mercury.

Control of the mercury pump was maintained by concurrently controlling the stainless steel 3-way valve (valve F) and the height of the mercury reservoir relative to the pump displacement cylinder. The chain provided a convenient way to quickly raise, lower, and set the reservoir to different heights. The pump technique, however, actually required a more delicate technique than what could be achieved with the chain. The reservoir needed to be raised and lowered a bit more smoothly for better control and reduced shock to the system.

One worrisome feature about the mercury pump was the capability to induce a "hammer effect" that would result when the flow of mercury was suddenly stopped inside the displacement cylinder. Careful control of the rate of flow of mercury was necessary to minimize the severity of the hammer effect.

6.2 Data Logging Method

The data logging software provided a convenient way to monitor and collect data during the experiments. During the authentic application this capability will be invaluable to successful operation.

6.3 System Leak Rate

Figure 6.1 illustrates a typical application of the procedure for determining the leak rate. In the Figure the abscissa represents time while the ordinate represents the system pressure. The linear regression of all the data points is represented by the line running through the data points and has the slope dimension (pressure/time). Specific parameters for this particular set of data were:

System volume: 1103 mL
 Temperature T: 298 K
 Slope (Pa/sec): 0.0195

The units for pressure and time are pascal and seconds (respectively) and, when applied to Equation 5.1 yield the system leak rate in units of $\mu\text{moles/sec}$. Thus, this experiment determined the leak rate of the system (without water vapor present) to be:

$$\begin{aligned} \text{Leak Rate} &= 0.0195 \frac{\text{Pa}}{\text{sec}} \times \frac{1104 \text{ mL}}{R \cdot 298 \text{ K}} && \text{Equation 6.1} \\ &= 0.00868 \mu\text{moles/sec} \end{aligned}$$

A system leak rate of this magnitude ($0.009 \mu\text{moles/sec}$) was frequently encountered on what was considered to be a vacuum tight system and did not appear to be too severe. If a leak rate of this magnitude were to exist on the actual setup during an authentic extraction operation, the deleterious effects on analytical quality, though measurable, would not be very serious.

It is estimated that a typical waste tank sample should contain about 40 mL of gas at atmospheric pressure. This amounts to about 0.0016 moles of gas. A leak rate of about $0.009 \mu\text{moles/sec}$ for 3600 seconds (1 hour extraction time) would yield an analytical error of about 2% in this case. Even this is a conservative estimate, however, since 1) it probably won't take a full hour to collect the gas samples (more like 30-40 minutes) and 2) the use of Cajon® type fittings rather than Swagelok® fittings will probably improve vacuum tight integrity of the system.

The design requirements (Reference 3) state that the maximum leak rate should be no more than 10^{-6} atm-cc/sec. This equates to $0.00004 \mu\text{moles/sec}$. If the

leak rate of the RGS system is controlled to this rate, there should be no detectable atmospheric contamination of the collected gases.

An analysis of the leak rate could be confirmed by observing the concentration of oxygen (O_2) in the collected gas sample. Analogous confirmations using nitrogen (N_2) and argon (Ar) would not be possible, however, due to the likelihood that these gases should already be present in significant quantity in the waste tank sample.

Though Figure 6.1 shows a general linear behavior between pressure and time, the graph also shows a curious regular oscillation of the pressure points about every three minutes or so. This oscillation was seen several times in different experiments and was determined to be caused by the system's reaction to the laboratory's central heating system. Apparently, the reactance of the pressure transducer, or the internal pressure of the system, or a combination of both, would change noticeably as a result of changes the temperature of the air in the laboratory due to the on/off cycling of the heater. To verify that this was the case, a digital readout of a thermocouple was set up in the same location as the system setup and indicated a 1-2°C oscillation of the laboratory climate at about the same frequency as the oscillations observed in Figure 6.1.

Figure 6.1: Leak Rate Determination

6.4 Mercury Pump Efficiency Determination

The mercury pump efficiency determination was used to see what pump displacement volume was adequate for the anticipated size of the retained gas sampler system. A pump displacement needed to be of the proportion necessary to achieve a good deal of the mass transfer (>99%) in a reasonable amount of time. Since the anticipated extractor volume is about 1000 mL, an adequate pump displacement was thought to be about 400-500 mL. The laboratory system was built with these anticipated volumes in mind and a pump displacement cylinder of 425 mL was constructed.

Figure 6.2 illustrates the time involved in evacuating a (dry) extractor system of 1103 mL with the 425 mL mercury displacement pump. The abscissa represents the time (minutes) involved in evacuating the extractor side of the system. The ordinate represents the extractor pressure (Pa). As the curve illustrates, greater than 99.9% (less than 100 Pa remaining out of 100,000 Pa) of the gas was removed from the extractor in about 45 minutes. This appeared to be a reasonable time frame for a system of these volumes. For perspective, the curve shown on the graph yielding greater than 99.9% air removal in about 10 minutes was performed using a mechanical vacuum pump.

Figure 6.3 illustrates the efficiency of the pump on a dry extractor (data points) relative to a theoretical extractor/pump system (straight line). Note that the ordinate is logarithmic. Again, the abscissa represents the time involved in evacuating the extractor side and the ordinate represents the pressure in the extractor. The straight line with negative slope (no data point indications) represents the theoretical pressure which would be expected from the operation of a perfect pump in a perfect (no leaks) system. The curve of individual data points represents the actual performance of the pump--each data point marking the point where the mercury pump was purged and reset for the next cycle.

The graph shows that for a dry system which contained no water for vapor out-gassing, the evacuation of the extractor from atmospheric pressure down to less than 300 Pa (99.7%) was achieved in 20 strokes of the pump (at about one stroke per minute). The performance of the pump was impressive since it seemed to perform almost perfectly in removing 99% of the air in the extractor before off-gassing and leaks became more noticeable.

Figure 6.4 illustrates the results a similar test to the one above but conducted on a wet system in which the extractor contained 300 mL water (ambient temperature). The efficiency of the pump appeared to have collapsed as the data points indicate a severely flattened time/pressure curve after about 10 minutes' time. Of course, it was not the efficiency of the pump that had fallen but rather the distillation of water which had compensated each pump cycle evacuation with its vapor pressure. The smooth curve (no data points) illustrates the performance of a theoretically perfect pump in the same type of environment. The agreement, therefore, between these two curves

shows that even in the presence of high water vapor pressure, the mercury pump continued to perform as well as any pump would be expected in this environment.

Figure 6.2: Mercury Displacement Pump Efficiency

Figure 6.3: Dry Extractor Evacuation Test

Figure 6.4: Wet Extractor Evacuation Test

6.5 Volume Determination

Figure 6.5 illustrates the application of Equation 5.3 in an experiment conducted to determine the volume of the extractor side of the setup. The concave downward curve shows the behavior of the resultant pressure of the extractor side as aliquots of air were let into the system. By solving for V_2 , the unknown volume in Equation 5.3, the extractor volume was determined after every aliquot. The average value of the V_2 is represented by the straight and level line in the graph which intersects the right-hand axis at 1103 mL.

To double check the accuracy of this determination a variation of Equation 5.2 was used.

$$\text{Theoretical Pressure } P_3 = \frac{P_1 V_1 + P_2 V_2}{V_1 + V_2} \quad \text{Equation 6.2}$$

This equation allows for the calculation of the theoretical pressure (P_3) based upon the initial pressure of the system (P_1) and a reasonable guess of the unknown volume (V_2). By using this equation in a software spreadsheet to repeatedly calculate the resultant pressure (P_3) after every aliquot of air, the shape of the curve could be made to exactly match the shape of the pressure curve which was actually acquired. This was done by selecting (trial and error) the correct value for V_2 . (This technique is actually a simplified spreadsheet method for conducting the technique in calculus known as "Newton's Method.")

Figure 6.6 illustrates this technique and shows that by using the value of 1103 for V_2 , a curve was generated (smooth, no data points) from Equation 6.2 and overlaid on the data points collected during the actual experiment. Because both curves appear to be identical, it seems that this is a valid test for the accuracy of the volume determination since the two curves (data points and smooth curve) were obtained independently, i.e. only the initial pressure point, (P_1) was common to both graphs--all other values determined independent.

Figure 6.5: Extractor Side Volume Determination

Figure 6.6: Volume Determination Validity Check (1103 mL)

6.6 Mass Transfer Simulation

The last experiment conducted on the laboratory setup was designed to simulate the mass transfer operation under conditions similar to those anticipated in the actual setup. Unfortunately it was not practical to construct a mock setup which replicated all the anticipated parameters of the actual setup and so an absolutely perfect simulation was not effected.

There were two parameters in particular which were not incorporated in the laboratory setup to note. 1) Deionized water was used instead of a waste tank simulant to generate the sample vapor pressure and 2) the sample in the extractor was not heated to tank temperature. However, since it was doubtful that even heated waste tank simulant sample could replicate the mechanism of gas release from tank waste accurately, the whole concept of employing simulated tank waste material in the laboratory setup was disregarded. However, by virtue of the colligative properties of aqueous solutions, the vapor pressure of the deionized water sample (at ambient temperature) was roughly equivalent to what would be expected for a high ionic strength waste tank sample heated to a temperature of about 40°C.

Figure 6.7 illustrates the pressure data collected when the extractor gas was transferred from the extractor to the collector. To begin, the whole system was evacuated to as low a pressure as the mechanical pump could achieve after about 20 minutes of operation, about 50 Pa. Next, 300 mL of water was sucked into the extractor to simulate the bulk of the waste tank sample and its corresponding vapor pressure. Finally, with valve E still open, a 40 mL aliquot of air was let into the extractor to simulate the gas phase component of the waste tank sample.

The first data point in Figure 6.7, the intersection of the two curves, represents a pressure measurement on both the extractor side pressure curve and the collector side pressure curve. The pressure of the extractor side and the collector side is equivalent because valve E is still open. After this, however, valve E was closed and from the first pump cycle to the last, the two pressure curves are seen to diverge--the top one being the pressure curve of the collector side and the bottom curve being the pressure curve of the extractor side.

In order to determine the success of this test (to determine whether mass transfer could in fact be conducted with this type of pump over a substantial vapor pressure) the pressure data of each acquisition was converted into millimole of gas using the ideal gas law.

Figure 6.8 illustrates similar curves shown in Figure 6.7 but graphed in units of millimole rather than pascal. Also shown in Figure 6.7 is the sum total amount of gas of the extractor and collector sides of the system. The graph shows that, from the start of the experiment to the finish, the total amount of gas in the system essentially remained constant throughout the experiment. This indicates that the operation of the mercury pump does not inadvertently add gas (air) to or subtract gas from the system through some overlooked

mechanism or route. The uniformity in the total amount of gas in the system from the start of the pumping operation to the finish, therefore, indicates that this mass transfer operation, as performed in this experiment, is wholly adequate for this purpose and demonstrates satisfactory performance.

Finally, included in Figure 6.7 is a line which represents the theoretical amount of gas originally let into the system. The overlap (agreement) of data points which represent the total amount of gas in the system and the line representing the theoretical amount of gas let in to the system, indicates that the mass transfer operation performs as well as theoretically expected.

In conclusion, it seems reasonable to assume that the design of the extractor system as described and tested in this report, is adequate for actual retained gas sampling of waste tank core samples.

Figure 6.7: Mass Transfer (Pressure)

Legend: ● Collector Side
■ Extractor Side

Figure 6.8: Mass Transfer (Millimole)

Legend:

- Extractor Side
- Collector Side
- × Sum of Extractor and Collector Sides

7.0 REFERENCES

1. Birney, K. R., Sherwood, D. J., 1993, Internal Memo to G. D. Johnson, *Retained Gas Sampler System Work Plan*, December 30, 1993, Westinghouse Hanford Company, Richland, Washington.
2. Hey, B. E., 1994, WHC-SD-WM-TP-201, *Test Plan For Cold Laboratory Setup of the RGS Extractor Simulator*, Westinghouse Hanford Company, Richland, Washington.
3. Wootan, D. W., 1994, WHC-SD-WM-FRD-018, Draft, Functional Design Criteria for the Retained Gas Sampler System, Westinghouse Hanford Company, Richland, Washington.

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8.0 ATTACHMENTS

Pressure Transducer Data Acquisition Program (BASIC)

10 SCREEN 9: KEY OFF: COLOR 7, 1: CLS

20 SCALE = 5

30 PRINT

40 PRINT " || WESTINGHOUSE HANFORD COMPANY || ";

50 PRINT

60 PRINT " || Process Chemistry Laboratory || ";

70 PRINT " || RETAINED GAS SEPARATION SYSTEM || B.E. Hey Scientist || ";

80 PRINT " || August 3, 1994 || ";

90 PRINT

100 PRINT " || DATA ACQUISITION PROGRAM FOR QE-DP41 PROCESS DISPLAY || ";

110 PRINT " || ";

120 PRINT " || MAIN MENU (enter number ...) || ";

130 PRINT " || ";

140 PRINT " || (1) Open COM1: and save data to file ACQUIRE.PRN || ";

150 PRINT " || ";

160 PRINT " || (2) Open COM2: and save data to file ACQUIRE.PRN || ";

170 PRINT " || ";

180 PRINT " || (3) Sampling frequency (Seconds) || ";

190 PRINT " || ";

200 PRINT " || (4) MONITOR PARAMETERS SETUP (Data Acquisition) || ";

210 PRINT " || ";

220 PRINT " || (5) RESET MENU || ";

230 PRINT " || ";

240 PRINT

250 PRINT

260 PRINT " Press <ESC> to exit to DOS";

270 K\$ = INKEY\$: IF K\$ = "" THEN 270

280 IF K\$ = CHR\$(27) THEN SYSTEM

290 IF K\$ = "1" THEN SOUND 1000, 1: LOCATE 12, 2: COLOR 10: PRINT " (1) Open COM1: and save data to file ACQUIRE.PRN

" : C1% = 1

300 IF K\$ = "2" THEN SOUND 1000, 1: LOCATE 14, 2: COLOR 10: PRINT " (2) Open COM2: and save data to file ACQUIRE.PRN

" : C2% = 1

310 IF K\$ = "3" GOTO 320 ELSE 400

320 SOUND 1000, 1: LOCATE 16, 2: COLOR 12: PRINT " (3) Sampling frequency: <0> INTERACTIVE / INTERVAL (SEC)"

330 LOCATE 16, 73: INPUT S\$

350 S% = VAL(S\$)

360 IF S% = 0 THEN LOCATE 16,36: COLOR 11: PRINT "INTERACTIVE " : SOUND 1000,1

370 IF S% > 0 THEN LOCATE 16,36: COLOR 11: PRINT USING "#### SECOND INTERVAL " : S%

380 IF S% < 1 THEN S% = 1

390 SOUND 1000,1

400 IF K\$ = "4" AND C1% = 0 AND C2% = 0 THEN BEEP: RUN

410 IF K\$ = "4" THEN SOUND 1000, 1: SOUND 2000, 1: GOTO 440

420 IF K\$ = "5" THEN RUN

430 GOTO 270

440 CLS

450 IF C1% = 1 THEN OPEN "COM1: 9600, O, 7, 1, RS, CS, DS" FOR RANDOM AS 1

460 IF C2% = 1 THEN OPEN "COM2: 9600, O, 7, 1, RS, CS, DS" FOR RANDOM AS 2

470 OPEN "ACQUIRE.PRN" FOR OUTPUT AS 3

480 COLOR 7, 1: CLS

490 PRINT

500 PRINT " || MONITOR PARAMETERS SETUP || ";

510 PRINT

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520 PRINT " ||
530 PRINT " ||
540 PRINT " ||
550 PRINT " ||
560 PRINT " ||
570 PRINT " ||
580 PRINT " ||
590 PRINT " ||
600 PRINT " ||
610 PRINT " ||
620 PRINT
" ||
" ||
630 LOCATE 5, 5: IF C1% = 1 THEN INPUT "Title for COM1: ....."; C1$ ELSE PRINT "COM1: INACTIVE"
640 LOCATE 6, 5: IF C2% = 1 THEN INPUT "Title for COM2: ....."; C2$ ELSE PRINT "COM2: INACTIVE"
650 LOCATE 7, 5: IF C1% = 1 THEN INPUT "Units of Measure for COM1: ...."; U1$ ELSE U1$ = "INACTIVE"
660 LOCATE 8, 5: IF C2% = 1 THEN INPUT "Units of Measure for COM2: ...."; U2$ ELSE U2$ = "INACTIVE"
670 LOCATE 10, 5: COLOR 14: PRINT "Press <+> or <-> to increase or decrease display range."
680 LOCATE 22, 46: COLOR 13, 0: PRINT "DATA ACQUISITION (Next Keystroke)": BEEP
690 LOCATE 23, 53: COLOR 14: PRINT "Press <ESC> to TERMINATE..."
700 K$ = INKEY$: IF K$ = "" THEN 700: SOUND 1000, .75: SOUND 2000, 1
710 IF K$ = CHR$(27) THEN RUN
720 COLOR 7, 0: CLS
730 LOCATE 1, 55: COLOR 3: PRINT "Press <ESC> to TERMINATE..."
740 COLOR 7, 0
750 PRINT
" ||
" ||
760 PRINT " || DATA ACQUISITION MODE || "
770 PRINT
" ||
" ||
780 PRINT " ||
790 PRINT " ||
800 PRINT " ||
810 PRINT " ||
820 PRINT " ||
830 PRINT " ||
840 PRINT " ||
850 PRINT " ||
860 PRINT " ||
870 PRINT " ||
880 PRINT " ||
890 PRINT " ||
900 PRINT " ||
910 PRINT " ||
920 PRINT " ||
930 PRINT " ||
940 PRINT " ||
950 PRINT " ||
960 PRINT
" ||
" ||
970 LINE (23, 84)-(281, 237), 7, BF
980 LINE (343, 84)-(601, 237), 7, BF
990 LINE (24, 85)-(280, 236), 1, BF
1000 LINE (344, 85)-(600, 236), 1, BF
1010 LOCATE 6, 4: COLOR 5: PRINT "COM1: "; C1$
1020 LOCATE 6, 44: COLOR 5: PRINT "COM2: "; C2$
1030 COLOR 3
1040 PRINT #3, "SAMPLE"; C1$; C2$
1050 PRINT #3, S%;"U1$;U2$
1060 DIM M1(64)
1070 DIM M2(64)
1080 FOR Z = 0 TO 64
1090 M1(Z) = 236: M2(Z) = 236

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1100 NEXT Z
1110 IF C1% = 1 THEN PRINT #1, "X01": I1$ = INPUT$(11, 1): IN1 = VAL(RIGHT$(I1$, 8))
1120 IF C2% = 1 THEN PRINT #2, "X01": I2$ = INPUT$(11, 2): IN2 = VAL(RIGHT$(I2$, 8))
1130 PRINT #3, A%; IN1; IN2: A% = A% + 1
1140 IF C1% = 1 THEN GOSUB 1900
1150 IF C2% = 1 THEN GOSUB 1940
1160 M1(64) = Y1
1170 M2(64) = Y2
1180 FOR Q = 0 TO 63
1190 M1(Q) = M1(Q + 1)
1200 M2(Q) = M2(Q + 1)
1210 NEXT Q
1220 IF SCALE = OLDSCALE THEN GOTO 1370
1230 IF SCALE > 9 THEN SCALE = 9
1240 IF SCALE < -4 THEN SCALE = -4
1250 LOCATE 7, 39: COLOR 7: PRINT "10";
1260 LOCATE 8, 39: COLOR 7: PRINT "9";
1270 LOCATE 9, 39: COLOR 7: PRINT "8";
1280 LOCATE 10, 39: COLOR 7: PRINT "7";
1290 LOCATE 11, 39: COLOR 7: PRINT "6";
1300 LOCATE 12, 39: COLOR 7: PRINT "5";
1310 LOCATE 13, 39: COLOR 7: PRINT "4";
1320 LOCATE 14, 39: COLOR 7: PRINT "3";
1330 LOCATE 15, 39: COLOR 7: PRINT "2";
1340 LOCATE 16, 39: COLOR 7: PRINT "1";
1350 LOCATE 17, 39: COLOR 7: PRINT "0";
1360 OLDSCALE = SCALE
1370 LINE (24, 85)-(280, 236), 1, BF
1380 LINE (24, 85 + 151 * .1)-(280, 85 + 151 * .1), 2
1390 LINE (24, 85 + 151 * .2)-(280, 85 + 151 * .2), 2
1400 LINE (24, 85 + 151 * .3)-(280, 85 + 151 * .3), 2
1410 LINE (24, 85 + 151 * .4)-(280, 85 + 151 * .4), 2
1420 LINE (24, 85 + 151 * .5)-(280, 85 + 151 * .5), 2
1430 LINE (24, 85 + 151 * .6)-(280, 85 + 151 * .6), 2
1440 LINE (24, 85 + 151 * .7)-(280, 85 + 151 * .7), 2
1450 LINE (24, 85 + 151 * .8)-(280, 85 + 151 * .8), 2
1460 LINE (24, 85 + 151 * .9)-(280, 85 + 151 * .9), 2
1470 LINE (344, 85)-(600, 236), 1, BF
1480 LINE (344, 85 + 151 * .1)-(600, 85 + 151 * .1), 2
1490 LINE (344, 85 + 151 * .2)-(600, 85 + 151 * .2), 2
1500 LINE (344, 85 + 151 * .3)-(600, 85 + 151 * .3), 2
1510 LINE (344, 85 + 151 * .4)-(600, 85 + 151 * .4), 2
1520 LINE (344, 85 + 151 * .5)-(600, 85 + 151 * .5), 2
1530 LINE (344, 85 + 151 * .6)-(600, 85 + 151 * .6), 2
1540 LINE (344, 85 + 151 * .7)-(600, 85 + 151 * .7), 2
1550 LINE (344, 85 + 151 * .8)-(600, 85 + 151 * .8), 2
1560 LINE (344, 85 + 151 * .9)-(600, 85 + 151 * .9), 2
1570 COLOR 10
1580 IF C1% = 1 THEN 1590 ELSE 1640
1590 IF ABS(IN1) < 10 ^ SCALE THEN COLOR 10 ELSE COLOR 12
1600 PSET (280, M1(64))
1610 FOR Z = 64 TO 0 STEP -1
1620 LINE -(Z * 4 + 24, M1(Z))
1630 NEXT Z
1640 IF C2% = 1 THEN 1650 ELSE 1700
1650 IF ABS(IN2) < 10 ^ SCALE THEN COLOR 10 ELSE COLOR 12
1660 PSET (600, M2(64))
1670 FOR Z = 64 TO 0 STEP -1
1680 LINE -(Z * 4 + 344, M2(Z))
1690 NEXT Z
1700 LOCATE 18, 4: PRINT " ";
1710 LOCATE 18, 4: COLOR 7: PRINT IN1; U1$
1720 LOCATE 18, 44: PRINT " ";
1730 LOCATE 18, 44: COLOR 7: PRINT IN2; U2$
1740 LOCATE 20, 4: COLOR 3: PRINT USING "SAMPLE NUMBER #####"; A%
1750 LOCATE 21, 4: COLOR 3: IF S% > 0 THEN PRINT USING "SAMPLING FREQUENCY (SEC) ####"; S% ELSE PRINT "INTERACTIVE

```

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```

SAMPLING";
1760 LOCATE 22, 4: COLOR 3: PRINT USING "SCALE <+> <->      1E+#"; SCALE - 1
1770 IF S% > 0 THEN 1780 ELSE 1880
1780 FOR STIME = 1 TO S%
1790 LOCATE 21, 29: COLOR 12: PRINT USING "####";(S% - STIME) + 1
1800 STIME$ = TIME$
1810 K$ = INKEY$
1820 IF K$ <> "" THEN GOSUB 1980
1830 IF STIME$ = TIME$ THEN 1830
1840 NEXT STIME
1850 SOUND 100,1
1860 GOTO 1110
1870 SOUND 100, 1: GOTO 1110
1880 K$ = INKEY$: IF K$ = "" THEN 1880 ELSE GOSUB 1980
1890 SOUND 1000, 1: GOTO 1110
1900 Y1 = 236 - ABS(IN1) * 151 / 10 ^ SCALE
1910 IF Y1 < 85 THEN Y1 = 85: LOCATE 6, 31: COLOR 12: PRINT "OVERSCALE";
1920 IF Y1 > 85 THEN LOCATE 6, 31: PRINT "      ";
1930 RETURN
1940 Y2 = 236 - ABS(IN2) * 151 / 10 ^ SCALE
1950 IF Y2 < 85 THEN Y2 = 85: LOCATE 6, 71: COLOR 12: PRINT "OVERSCALE";
1960 IF Y2 > 85 THEN LOCATE 6, 71: PRINT "      ";
1970 RETURN
1980 IF K$ = CHR$(27) THEN RUN
1990 IF K$ = "+" THEN SCALE = SCALE + 1
2000 IF K$ = "-" THEN SCALE = SCALE - 1
2010 RETURN

```