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COMPUTERIZED 50 LITER VOLUME CALIBRATION SYSTEM (U)

by

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ABSTRACT

A system has been designed for the Savannah River Site that will be used to calibrate product shipping containers. For accountability purposes, it is necessary that these containers be calibrated to a very high precision. The Computerized 50 Liter Volume Calibration System (CVCS), which is based on the Ideal Gas Law (IGL), will use reference volumes with a precision of no less ±0.03%, and helium to calibrate the containers to have a total error of no greater than ±0.10%. A statistical interpretation of the system has given a theoretical total calculated error of ±0.08%. Tests with the system will be performed once fabrication is complete to experimentally verify the calculated error. Since the total error was calculated using the worst case scenario, the actual total error should be significantly less than the calculated value. The computer controlled, totally automated system is traceable to the National Institute of Standards and Technology. The design, calibration procedure, and statistical interpretation of the system will be discussed.

INTRODUCTION

The LP-50 Shipping Container is used by the Department of Energy's (DOE's) Savannah River Site to ship elemental tritium to other DOE sites. For accountability purposes, it is necessary that these containers be calibrated to a very high precision. The cylinders now in use were calibrated in the late 1970's and have a total error of approximately ±1.9%. In order to comply with AEC Technical Information Division Manual TID-26298[1] and DPSPWD-84-313, it was requested that a system be designed that had a total error of no greater than ±0.10% at a 0.95 probability level.

BACKGROUND

The Computerized 50 Liter Volume Calibration System (CVCS) is based on the Ideal Gas Law (IGL) whose equation is written as:

$$PV = nRT \quad .... (1)$$

where P is the pressure of the gas, V is the volume of the container, n is the number of moles of gas, R is the Universal Gas Constant, and T is the temperature of the gas. Because the calibration calculations for the CVCS are directly derived from the IGL, three parameters of the system, pressure, temperature, and volume, must be monitored closely. Helium, which acts as a perfect gas at low pressures will be used to perform the calibrations.

SYSTEM ARCHITECTURE

Overall System

The CVCS consists of several major components including the pressure measurement devices, the piping system, the reference volumes, and the instrumentation (See Figure 1). The piping system, reference volumes, and some instrumentation will be located in a Tenney Constant Temperature Chamber whose temperature will be controlled to within ±0.1°C. Following is a brief description of each of the major components.

Pressure Measurement Devices

Two pressure measurement devices are located in the system. They are an MKS Baratron High Accuracy Sensor Head and a Paroscientific Digiquartz Intelligent Transmitter. The MKS Baratron will be used to measure the pressure of the system in its evacuated state. The sensor has an accuracy of ±0.05%. While the accuracy of the MKS Baratron is important, it is not as vital as the accuracy of the

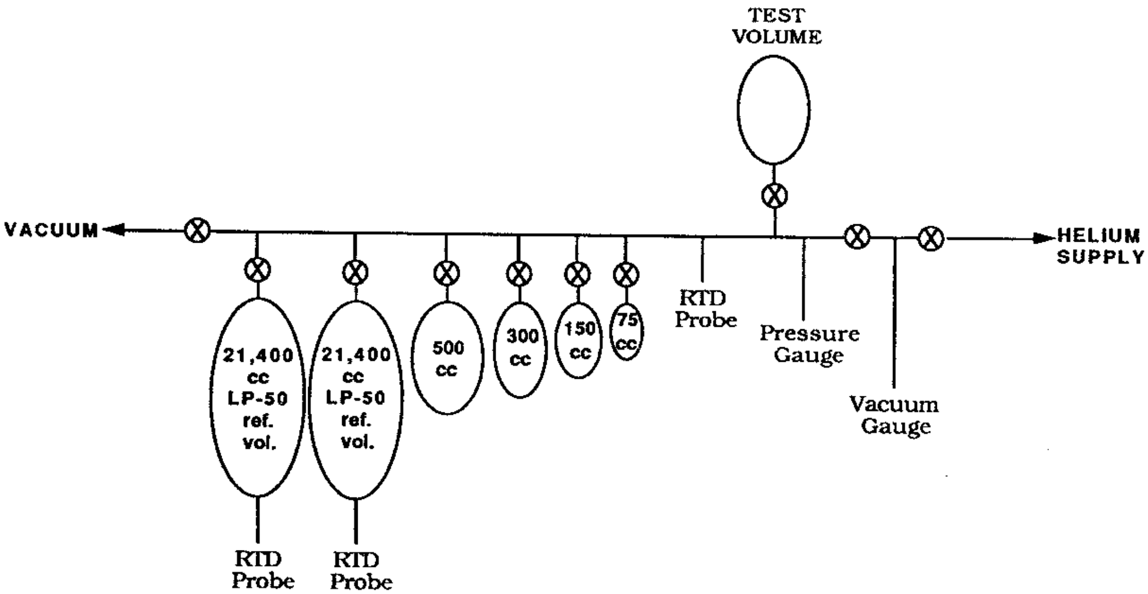

Figure 1

Paroscientific Digiquartz Intelligent Transmitter. The Paroscientific will make all pressure measurements during a test run. These measurements will be used in the volume calculations, therefore a higher accuracy is required. The accuracy of this device is $\pm 0.01\%$. Due to the error propagation in the system, a device with a lower accuracy (i.e. greater than $\pm 0.01\%$) would cause the total calculated error to be outside the acceptable range.

Piping System

The piping system manifold is constructed of 1/4" 316 stainless steel tubing. Located on the manifold are various valves and cylinders. The use of the IGL required that the amount of helium in the system remain constant throughout one measuring cycle. Therefore, Whitey Constant Volume Ball Valves were used on the main manifold and on each of the reference volumes.

LP-50 and Hoke Reference Volumes

There are two types of cylinders located on the manifold: the LP-50 Reference Volumes and Hoke sampling cylinders.

The Hoke sampling cylinders will not be used to calibrate the LP-50 shipping containers, but will be used to calibrate volumes of less than 10 liters. The cylinders will be calibrated to have a total error of no greater than ±0.03% and will be traceable to the National Institute of Standards and Technology.

The LP-50 Reference Volume was designed specifically for the CVCS to calibrate the LP-50 shipping container. Each reference volume was fabricated of schedule 80 316 stainless steel pipe with 1/2" thick end plates. The use of heavy walled material reduced the amount of flex on the reference volume to a negligible level in the evacuated state and hence reduced calibrated volume drift to a negligible level. The reference volumes are of all welded construction (using full penetration welds). This was done to limit permeability and absorption of gas in the measuring system. At the calibration pressures, there will be negligible absorption of gas into the metal. Platinum Resistance Temperature Detectors were inserted into each reference volume so that a constant temperature could be verified within the system. Each reference volume is approximately 21.4 liters. There are two LP-50 Reference Volumes located on the manifold.

Instrumentation

The CVCS is operated by a Hewlett-Packard Model 305 Technical Computer System. The computer controls the opening and closing of valves. The computer will also read and record values for the pressure and vacuum gages, temperature probes, the temperature of the constant temperature chamber, and will perform all calculations and statistical analyses.

CALIBRATION OF CONTAINERS

In the calibration system, a ratioing system is used in conjunction with the IGL to relate a known and unknown volume (the LP-50 Reference Volume and the LP-50 shipping container respectively when calibrating the LP-50 shipping container). Initially, the entire system is evacuated. The computer, upon being given the approximate volume of the container to be calibrated by the operator, will select the appropriate sequence of reference volumes for ratioing (for this discussion the volume to be calibrated will be an LP-50 shipping container and the reference volume selected will be the LP-50 Reference Volume). The selected reference volume(s) and the unknown tubing are then filled to approximately 10 psia with helium. Once equilibrium is reached, the pressure, P_1, is read and recorded by the computer. The gas will then be allowed to expand into the unknown volume. Once equilibrium is reached, the pressure, P_2, is read and recorded by the computer. By combining these values with the IGL, the following equation is reached:

$$P_1(V_1 + V_2) = P_2(V_1 + V_2 + V_3) \text{ .... (2)}$$

V_1 = LP-50 Reference Volume (known)
V_2 = tubing volume (unknown)
V_3 = LP-50 shipping container (unknown)

Since the temperature is held constant throughout a test run, its values will cancel and it is therefore eliminated from the equation.

In order to derive a second equation, a reverse ratio is performed. The system is evacuated. Then the LP-50 shipping container, V_3, and the unknown tubing, V_2, are filled with helium to approximately 10 psia. Once equilibrium is reached, P_3 is read and recorded by the computer. The gas then expands into the LP-50 Reference Volume. After equilibrium is reached, P_4 is read and recorded by the computer. This second equation is written as:

$$P_3(V_2 + V_3) = P_4(V_1 + V_2 + V_3) \text{ .... (3)}$$

Equations (2) and (3), with two unknowns (V_2 and V_3), can be reduced to the following equation, in which V_2 has been eliminated:

$$V_3 = V_1 \frac{P_3[P_1 - P_2]}{P_1[P_3 - P_4]} \text{ .... (4)}$$

From this equation the volume of the LP-50 shipping container can be calculated directly.

When calibrating a 50 liter container, such as the LP-50 shipping container, there is a built in check point that verifies the accuracy of the calibration. Using the calculated value for the LP-50 shipping container, the internal tubing volume can be determined from equation (2) or (3). As shown in Figure 1, there is a second LP-50 Reference Volume. The helium in the system will now be allowed to expand into this cylinder and the equalized pressure, P_5, is read and recorded. Since the volume of this cylinder is known, a final theoretical expanded pressure can be calculated from the following equation:

$$P_5 = P_4 \frac{(V_1 + V_2 + V_3)}{(V_1 + V_2 + V_3 + V_4)} \quad .... \ (5)$$

in which V_4 is the volume of the second LP-50 Reference Volume. If the final actual expanded pressure does not fall within $\pm 0.05\%$ of the calculated pressure, the test is thrown out. The allowable error of $\pm 0.05\%$ was obtained from the following:

$$\text{total error} = (\text{error}_1^{\,2} + \text{error}_2^{\,2})^{1/2} \quad .... \ (6)$$

Since the maximum allowable total error is $\pm 0.10\%$ and the calculated total error is $\pm 0.08\%$ then:

$$0.10 = (0.08^2 + \text{error}_2^{\,2})^{1/2} \quad .... \ (7)$$

and

$$\text{error}_2 = 0.06\%$$

It was desired that the total system error remain below $\pm 0.10\%$, therefore $\pm 0.05\%$ was chosen.

STATISTICAL ANALYSIS

To determine the theoretical error, or uncertainty, of the system the Propagation of Errors method was used. The following modified version of equation (4) is used to obtain this error (this time temperature is included because it does have an uncertainty associated with it):

$$V_3 = V_1 \frac{P_3 T_4 [P_1 T_2 - P_2 T_1]}{P_1 T_2 [P_3 T_4 - P_4 T_3]} \quad .... \ (8)$$

To find the maximum standard deviation, σ, the partial derivative of the function is taken with respect to each variable with an uncertainty associated with it. The following equation is the base equation for performing this task:

$$\sigma = \left\{ \left[\frac{\partial F}{\partial X}\right]^2 \sigma_x^2 + \left[\frac{\partial F}{\partial Y}\right]^2 \sigma_y^2 + 2\left[\frac{\partial F}{\partial X}\frac{\partial F}{\partial Y}\right]\sigma_{xy} \right\}^{1/2} \quad \text{.... (9)}$$

Below is a sample derivative showing the function V_3 with respect to P_2:

$$\frac{\partial V_3}{\partial P_2} = \frac{-T_1P_3T_4V_1\,(P_1T_2[P_3T_4 - P_4T_3]) - 0}{(P_1T_2[P_3T_4 - P_4T_3])} \quad \text{.... (10)}$$

Once all the terms are solved as shown above, the nominal value for each variable (and its associated error) is plugged into the equation and the theoretical standard deviation is determined. It should be noted that the third term in equation (9), the covariance of the system, becomes negligible when the nominal values are inserted into the equation. The theoretical standard deviation for this system when calibrating a 50 liter container is is $\pm$39.77cc or $\pm$0.08%, which falls well within the required range of $\pm$0.10%.

SUMMARY

In order to meet the Department of Energy's requirements for the accountability of tritium, it is necessary to have high accuracy calibration equipment available. At the Savannah River Site, a system, based on the Ideal Gas Law, will be available with the capability to calibrate large containers to an accuracy of no less than $\pm$0.08% at a 0.95% probability level.

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REFERENCE

1 Jaech, J. L., "Statistical Methods in Nuclear Material Control", TID-26298, 1973.