HELIUM LEAK TESTING OF A RADIOACTIVE CONTAMINATED VESSEL

UNDER HIGH PRESSURE IN A CONTAMINATED ENVIRONMENT

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ABSTRACT

Argonne National Laboratory-West (ANL-W) is located 35 miles west of Idaho Falls, Idaho at the Idaho National Engineering Laboratory (INEL). The INEL boasts of producing the first electrical power generated by a nuclear reactor. The INEL is devoted to the research and development of all aspects concerning the nuclear industry. Safety is the number one concern at the INEL and at ANL-W.

The Argonne West site is operated by the University of Chicago under a contract with the Department of Energy. With the shut-down of Experimental Breeder Reactor II at ANL-W, research and development has evolved from advanced reactor design to the safe handling, processing, packaging and transporting spent nuclear fuel and nuclear waste. New methods of processing spent fuel rods from reactors and transforming contaminated material into acceptable waste forms are now in development.

Storage of nuclear waste is a high interest item. ANL-W is participating in the research of safe storage of nuclear waste, with the Waste Isolation Pilot Plant (WIPP) site located in New Mexico the repository.

The vessel under test simulates gas generated by contaminated material stored underground at the WIPP site. The test vessel is 90 percent filled with a mixture of contaminated material and salt brine (from the WIPP site) and pressurized with nitrogen with a tag of one percent helium to a pressure of 2500 psia. Test acceptance criteria is leakage of not more than 1 x 10^{-7} cc/seconds at 2500 psia.

The bell jar method is used to determine leakage rate using a MSLD. The efficient MSLD and an aluminum bell jar replaced a costly and elaborate time consuming pressure decay test set-up. Misinterpretation of test criterion data caused lengthy delays, resulting in the development of a unique procedure. Reevaluation of the initial intent of the test criteria resulted in leak tolerances being corrected and test efficiency improved.

DISCLAIMER

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SUMMARY

1. INTRODUCTION

Argonne National Laboratory-West is actively involved in nuclear research.

2. THE EXPERIMENT

The Gas Generation Experiment (GGE) is being performed to generate data for the safe storage of nuclear waste.

3. DESCRIPTION OF CONTAINER

A unique container was fabricated to test the feasibility of underground storage of nuclear waste.

4. DESCRIPTION OF DECAY TEST

Preliminary leak testing involved a time consuming pressure decay test. This test was replaced with a Helium Mass Spectrometer leak detector system (MSLD) which significantly reduced the testing time.

5. DESCRIPTION OF MSLD TEST SET UP

Modifications to the containment box were made to accept the MSLD for leak testing, precautions were taken to protect personnel from radioactive contamination.

6. LEAK TESTING WITH THE MSLD

A unique cover gas system was developed to eliminate high levels of helium background in the glove box. The MSLD proved itself superior to the pressure decay test system.

7. EVALUATION OF TEST CRITERIA

Discovery of a misinterpretation of the test criteria and overlooked pretest data, lead to the improved efficiency and successful completion of leak testing the GGE containers.

8. CONCLUSION

Close attention to details prior to leak testing cannot be sacrificed to meet a schedule.

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Storage of nuclear waste is a high-interest item. ANL-W is participating in the research of safe storage of nuclear waste, with the Waste Isolation Pilot Plant (WIPP) site located in New Mexico, the repository.

The test vessel is filled to 90% of its capacity with a mixture of waste and salt brine. It is then pressurized to 2500 psia. Test acceptance criterion is leakage of no more than 1×10^{-7} cc/second at 2500 psia.

THE EXPERIMENT

The gas generation experiment (GGE) simulates gases generated by contact-handled transuranic (TRU) waste stored at the WIPP site in Carlsbad, New Mexico. The entire test is contained within an argon filled glove box that is maintained at three inches water gauge negative with respect to ambient atmosphere. The argon atmosphere is purified and maintained at less than 50 ppm 0_2.

The test container is filled to 90% capacity with a mixture of contact-handled TRU waste and salt brine (from the WIPP site). Microbes that are found in the salts at the WIPP site are added to the test container before the container is sealed. The container is then sparged for 24 hours with a mixture of 99% nitrogen/one percent helium. The sparging process is used to clean out the mixture of gasses present in the container and give the test a baseline to start from. The one percent helium in the sparging process is used as a tag gas for the Mass Spectrometer Leak Detector (MSLD) leak testing. The test container is then pressurized with the 99% nitrogen/one percent helium gas to 2500 psia. This simulates the litho-static pressure of the WIPP Repository.

Gas samples are taken through a series of valves on top of the test container at predetermined intervals and analyzed with a gas chromatograph. Pressure and temperature are monitored continuously with instrumentation. Data are collected and stored by computer.

DESCRIPTION OF CONTAINER

The GGE container (see Figure 1) is constructed of Hastelloy C-276, a super corrosion resistant material with a high chrome-molly content. The container stands approximately 20 inches tall and has a diameter of 6.75 inches, with a volume of two gallons. The top of the container has a half-diamond-shaped groove machined into it for the seal ring to sit in. The lid of the container is in the shape of a top hat, with a half-diamond-shaped groove machined into the bottom for the seal ring to sit in. The material of the lid is also constructed of Hastelloy C-276. The top of the lid has penetrations to accept the various fittings of the valve tree (see Figure 2).

The seal ring is constructed of the same material as the container and the cross-section of the seal ring is in the shape of a diamond to completely fill the sealing ring groove. The device used to secure the container lid to the container is a "C"- shaped clamshell, split in half with 10 torquing bolts on both sides. A torque of 55 foot-pounds to 70 foot-pounds is applied to the torquing bolts to seal the container.

DESCRIPTION OF DECAY TEST

Initially, the GGE containers were to have a nitrogen-filled headspace, with a pure inert gas as the pressurizing medium. The decision to leak test the container using the pressure decay method was made early in the development of the experiment. A high-vacuum chamber that would house the test container with the attached valve tree assembly was chosen. The equipment for this test was procured and tested. The generation of a procedure that produced reliable test results followed.

The procedure called for a 48-hour vacuum leak test to produce a reliable, repeatable leak rate. The 48 hours required for the test seemed high, in the fact that up to 20 containers may have to be leak tested. If a container was found to have a leak, this type of test has no provisions for identifying the leak site. Upon evaluation of the test data, it was suggested that a MSLD could be used to perform the leak test in less than one hour if a helium tracer gas could be used in the head space without affecting the GGE. In addition to using an MSLD for the overall leak test, the container seal and the value tree assembly could be initially leak tested with a detector probe to identify and isolate leaks before the overall leak test was performed.

DESCRIPTION OF MSLD TEST SET UP

The leak detector used for the leakage test is a contra-flow helium mass spectrometer. This type of MSLD was chosen over the conventional flow type of MSLD because of its ability to start leak testing at a high port pressure. This particular model could be brought on line at 100 millitorr to start leak testing. This eliminated lengthy pump down times needed in the high vacuum port pressure conventional flow MSLD.

The two leak-test methods used for this test are the detector probe and the pressure vacuum method. Two separate systems were needed to provide leak testing (see Figure 3). The MSLD is connected to a manifold that has a pressure-sensing device set at 100 millitorr. This pressure-sensing device controls a pneumatic operated valve that closes when increasing pressure reaches 100 millitorr. This system is used to control any air leakage into the glove box in case the leak test operator vents the MSLD without valving it out of the system. High Efficient Particulate Air (HEPA) filters are in line with both the detector probe and the outlet of the service well. These are used to protect the MSLD from any loose radioactive particles in the system. A calibration port is downstream of the service well HEPA filter. The rouging pump is vented to suspect exhaust to keep the room atmosphere free of any radioactive contaminates (should a HEPA filter fail), and argon pumped out of the glove box during leak testing.

An aluminum bell jar was built to place over the service well to perform the pressure vacuum test. The bell jar stands approximately 22 inches high and has a diameter of 12 inches. A urethane rubber gasket is used to seal the bell jar around the service well. The bell jar has a calibration port and a vent port built near the top edge. The top of the bell jar has a pressure relief valve should the test container vent during leak testing.

LEAK TESTING WITH THE MSLD

Preliminary testing with the MSLD and the bell jar was done to gather base line data. These data included pump down times for the bell jar and response time for the MSLD.

Pump down times for the bell jar using the 7.0 cfm rouging pump to reach a pressure of 100 millitorr were under five minutes. Response time of the MSLD to detect a leak rate of 1×10^{-8} std cc/second He was under three seconds. The background helium rate was in the area of 3×10^{-9} std cc/second He, depending on the length of the test. These data on the surface appeared satisfactory. The test now could be completed in a fraction of the time that was needed to perform the pressure-decay test previously developed.

Modifications proceeded to disassemble the pressure-decay testing apparatus on the glove box and a new system for the MSLD leak testing was fabricated. Information disseminated to the leak test technicians stated that a leak rate of not more than 1×10^{-7} std cc/second He was acceptable. With a MSLD with a sensitivity of 2×10^{-10} std cc/second He and a response time of less than four seconds, the only concern was a high background level. The glove box atmosphere would be pure argon so there was little concern of a high helium background.

When modifications were completed on the glove box for the new MSLD leak test system, baseline data collection started. Pump down times corresponded to the mock-up testing. Sensitivity testing held true and background levels were acceptable. The only thing overlooked was the fact the tracer gas was one percent helium not the 100 percent helium with which the preliminary testing had been done. Calculations were made to determine the new acceptable leak rate readout as in the following formula:

$$\text{Leak rate} = \frac{\text{std calibrator x leak rate x percent helium}}{\text{actual calibrated leak rate x percent helium}}$$

$$\text{Leak rate} = \frac{2.6 \times 10^{-8} \text{ std cc/second He} \times 1 \times 10^{-7} \text{ std cc/second He} \times 1\% \text{ He}}{2.6 \times 10^{-8} \text{ std cc/second} \times 100\ \% \text{ He}}$$

$$\text{Leak rate} = 1 \times 10^{-9} \text{ std cc/second He}$$

The acceptable leak rate was now not more than 1×10^{-9} std cc/second He. This was still one decade greater than the sensitivity of the MSLD, but what would be the background in the argon atmosphere glove box?

When the glove box was filled with argon prior to the introduction of any radioactive material into the glove box, the leak-test system testing began with the new leak test criteria. Pump down times remained the same and the background levels were in the area of 2×10^{-10} std cc/second. Sensitivity and response time of the MSLD and associated leak-test system was excellent.

Because of the acceptable test criteria being just one decade above the sensitivity of the MSLD, the decision to increase the sensitivity of the MSLD was made. The diffusion pump temperature was adjusted to minimum and the calibration adjustment to near its maximum. The sensitivity was then calculated as in the formula below:

$$\text{Sensitivity} = \frac{\text{std calibrator x readout sensitivity range x percent helium}}{\text{actual calibrator leak meter reading x percent helium}}$$

The MSLD was nominally adjusted to display a readout of 1×10^{-7} std cc/second He with a calibrated standard of 2.6×10^{-8} std cc/second He. This resulted in a MSLD sensitivity as below.

$$\text{Sensitivity} = \frac{2.6 \times 10^{-8} \text{ std cc/second} \times 2 \times 10^{-10} \text{ std cc/second} \times 100\%}{1 \times 10^{-7} \text{ std cc/second} \times 100\%}$$

$$\text{Sensitivity} = 5.2 \times 10^{-11} \text{ std cc/second He}$$

With the sensitivity adjusted to this level the MSLD does not read out directly. All leak rates now must be calculated with the formula below

$$\text{Leak rate} = \frac{\text{std calibrator x actual test leak meter reading x percent helium}}{\text{posttest actual calibrator leak meter reading x percent helium}}$$

The first test container was filled with cold waste and brine, only. The container was lowered into the service well, the lid was secured and the lid bolts torqued. The test container was sparged for a period of 24 hours with a mixture of 99% nitrogen /one percent helium. The container was pressurized to approximately 50 psia and a detector probe test was performed. No gross leaks were found. The test container was then pressurized to approximately 2500 psia. The bell jar was placed over the test container complete with valve tree attached. The MSLD was calibrated with a standard of 2.6×10^{-8} std cc/second He. The readout was adjusted to a span of 3.4×10^{-8} std cc/second He. The sensitivity at this setting is:

$$\text{Sensitivity} = \frac{2.6 \times 10^{-8} \text{ std cc/second x } 2.0 \times 10^{-10} \text{ std cc/second x } 100\% \text{ He}}{3.4 \times 10^{-8} \text{ std cc/second x } 100\ \% \text{ He}}$$

$$\text{Sensitivity} = 1.53 \times 10^{-10} \text{ std cc/second He}$$

The test container was pumped down to 32 millitorr in 15 minutes. The MSLD readout indicated 1.4×10^{-7} std cc/second He. The MSLD readout was monitored for 40 minutes. During that time, the indicated leak rate fell to 1×10^{-8} std cc/second He. Calculations were made to check actual leak rate as follows:

$$\text{Leak rate} = \frac{2.6 \times 10^{-8} \text{ std cc/second x } 1.0 \times 10^{-8} \text{ std cc/second x } 100\% \text{ He}}{3.4 \times 10^{-8} \text{ std cc/second x } 1\% \text{ He}}$$

$$\text{Leak rate} = 7.64 \times 10^{-7} \text{ std cc/second He}$$

The corrected leak rate was still almost a decade away from meeting the leak test criteria.

It was determined that a high background was present and more pumping time would produce a more accurate leak rate. At 1600 hours the test was left to continue to pump down overnight. At 0725 hours the next morning the pressure in the bell jar was 29 millitorr and the MSLD read out indicated a leak rate of 2×10^{-9} std cc/second He.

A posttest of the MSLD produced the following data: Background 1×10^{-9} std cc/second He, full scale readout of 3.4×10^{-8} std cc/second He (with the 2.6×10^{-8} std cc/ second He standard).

Actual leak rate was calculated as follows.

Subtracting the background of 1.0×10^{-9} std cc/second from the total read out of 2.0×10^{-9} std cc/second equals the total indicated leak rate of 1.0×10^{-9} std cc/second He.

$$\text{Actual leak rate} = \frac{2.6 \times 10^{-8} \text{ std cc/ second } \times 1.0 \times 10^{-9} \text{ std cc/second} \times 100\ \%\ \text{He}}{3.4 \times 10^{-8} \text{ std cc/ second } \times 1\%\ \text{He}}$$

Actual leak rate = 7.6×10^{-8} std cc/second He

This leak rate is below the maximum test criteria but does not allow for much error. The background in the glove box was much higher than expected. After some inquiries it was discovered that one of the processes to ready the test container for the GGE had a loose fitting and leaked a large quantity of helium into the glove box.

It was suspected that the urethane rubber gasket used to seal the bell jar around the service well might have a small amount of leakage or that the helium in the glove box was permeating through the gasket material. A test of the gasket material revealed that the urethane rubber did indeed permeate helium in approximately 30 seconds.

The bell jar gasket was removed from the glove box and cleaned. Upon returning the gasket to the glove box, more tests were performed with the bell jar empty to determine the amount of background and the time required to pump out the helium to perform a leak test. Preliminary background testing indicated that the amount of helium in the glove box would hinder the leak test efficiency.

Because of the use of a contra flow type MSLD, it was suspected that the large amount of helium released into the glove box had contaminated the suspect exhaust ducts and helium was back streaming into the MSLD. With the permission from Radiation Safety personnel, the rouging pump exhaust was removed from the suspect duct. Background immediately dropped from 5 x 10^{-9} std cc/second to below zero.

The idea of having the MSLD disconnected from the suspect exhaust was not acceptable from a safety standpoint. Radiation contamination was not a major concern because of the HEPA filters integral to the leak test system. The inert atmosphere in the glove box posed more of a threat to the safety of personnel if the rouging pump was allowed to pump argon into the room. The need to reduce background levels with some other method had to be addressed.

Because the test container was in a glove box with an argon atmosphere and the addition of argon to the glove box would not have an adverse effect on the experiment, it was proposed to envelope the bell jar assembly with a plastic bag and purge the bag with pure argon to isolate the bell jar gasket from the helium-contaminated argon atmosphere.

A large heavy-weight plastic bag was cut to size and weighted around the opening to hold it in place during the purging process. The bag was put into the glove box, placed over the bell jar, and testing began with the bell jar empty. The bell jar was pumped down and the MSLD placed in test. A plastic tube was connected between a pure argon inlet fitting and the plastic bag. With the purge of fresh argon around the bell jar for a period between 20 to 30 minutes, background levels dropped significantly. Preliminary testing showed a decrease in the background level in the order of 5 x 10^{-8} std cc/ second He. This amount of background could easily mask the true leak rate of the test container and cause the test to fail.

The second test container was readied for the GGE and placed in the service well for leak testing. The detector probe method was used to look for gross leaks around the valve tree and the seal. The container leaked around the seal and was retorqued numerous times before passing the detector probe test. The bell jar was placed over the test container and pumped down to place the MSLD in test mode. The test container failed the leak test, was retorqued and leak tested three times before being put aside to continue with another container.

The third container was readied for the GGE and placed in the service well. The detector probe test was performed and no gross leaks were found. The bell jar was placed over the test container and pumped down to place the MSLD in test mode. The container failed the leak test the first, but after retorquing passed the seal test on the second try with the use of the plastic bag and pure argon purge. The effectiveness in the reduction of background using the plastic bag and argon purge was proven during this test.

EVALUATION OF TEST CRITERIA

During the testing of the third container, evaluations were being made on the data collected from the first two containers that were leak tested. It was discovered that a misinterpretation of the leakage test criteria had been made. The Technical Requirements Document was reviewed and the test criteria stated a leakage rate of not more than 1 x 10^{-7} cc/second at 2500 psia. The leak rate that was being used was 1 x 10^{-7} std cc/second He or 1 x 10^{-7} cc/second He at 14.7 psia. Calculations were made to determine the actual leak rate criteria using Boyle's law, below.

P1V1 = P2V2

P1 = 2500 psia or 170 atmospheres (2500/14.7 = 170)

V1 = 1cc/second

P2 = 14.7 psia, or one atmosphere

V2 = P1V1/P2

V2 = (2500psia x 1cc/ second) / 14.7 psia

V2 = 170 cc/second at (14.7 psia or one atmosphere)

V2 = 170 std cc/second

then 1 $x10^{-7}$cc/second at 2500psia = 170 $x10^{-7}$ std cc/second at 2500 psia

or 1.7 $x10^{-5}$ std cc/ second at 2500psia.

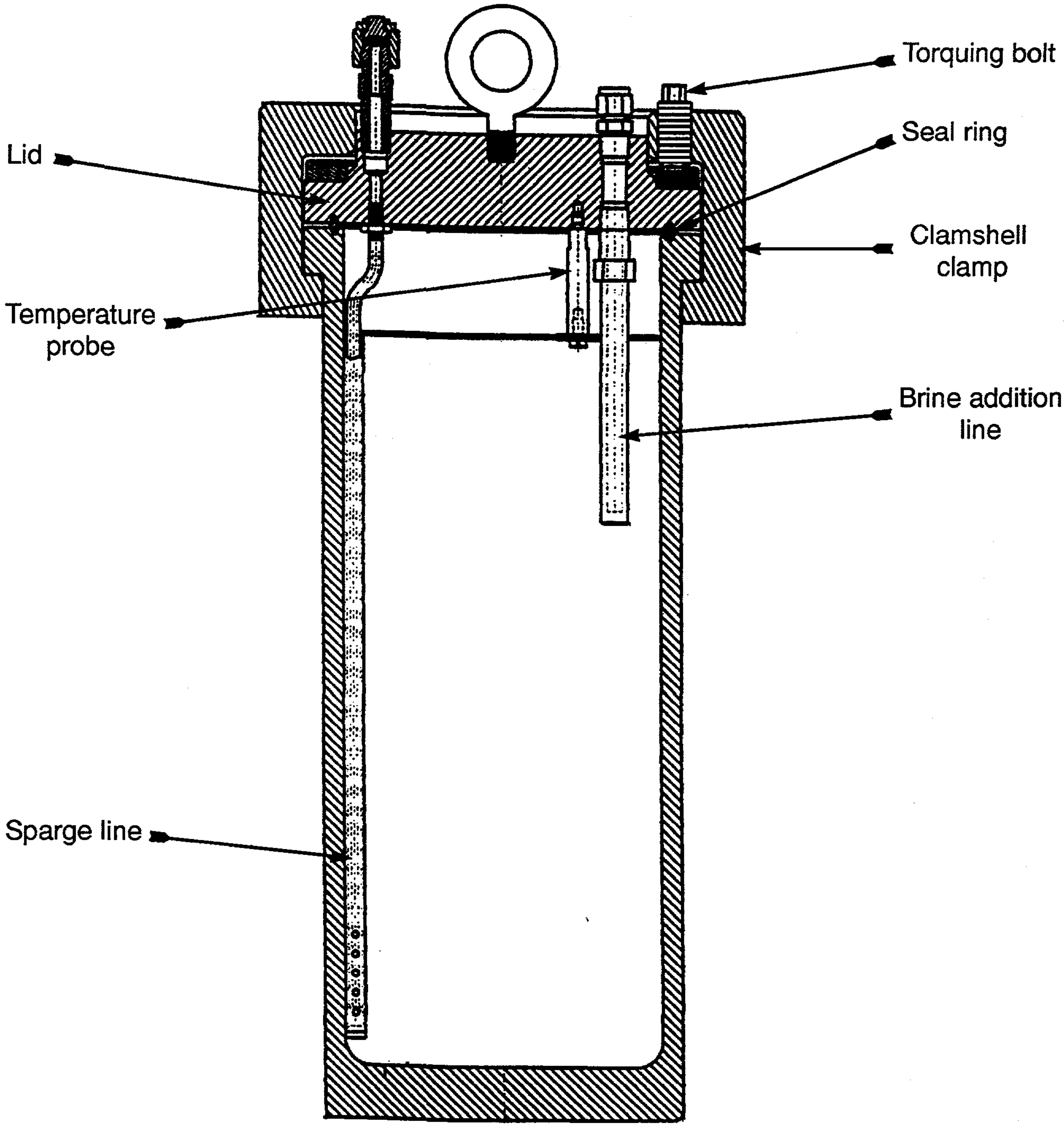
This discovery now relaxed the leak test criteria by nearly two orders of magnitude. With the new test requirements the problems with background were easily overcome.

In approximately the same time frame it was also discovered that a dry lubricant had been substituted on the torquing bolts during the pressure decay testing. It was feared that outgassing of the liquid-based lubricant would adversely affect the leak test. The dry lubricant used was found to not give a consistent torque on each bolt. When the original specified lubricant was used on the torquing bolts, subsequent leak tests were easily passed even with higher-than-normal backgrounds and the completion of sixteen test containers followed.

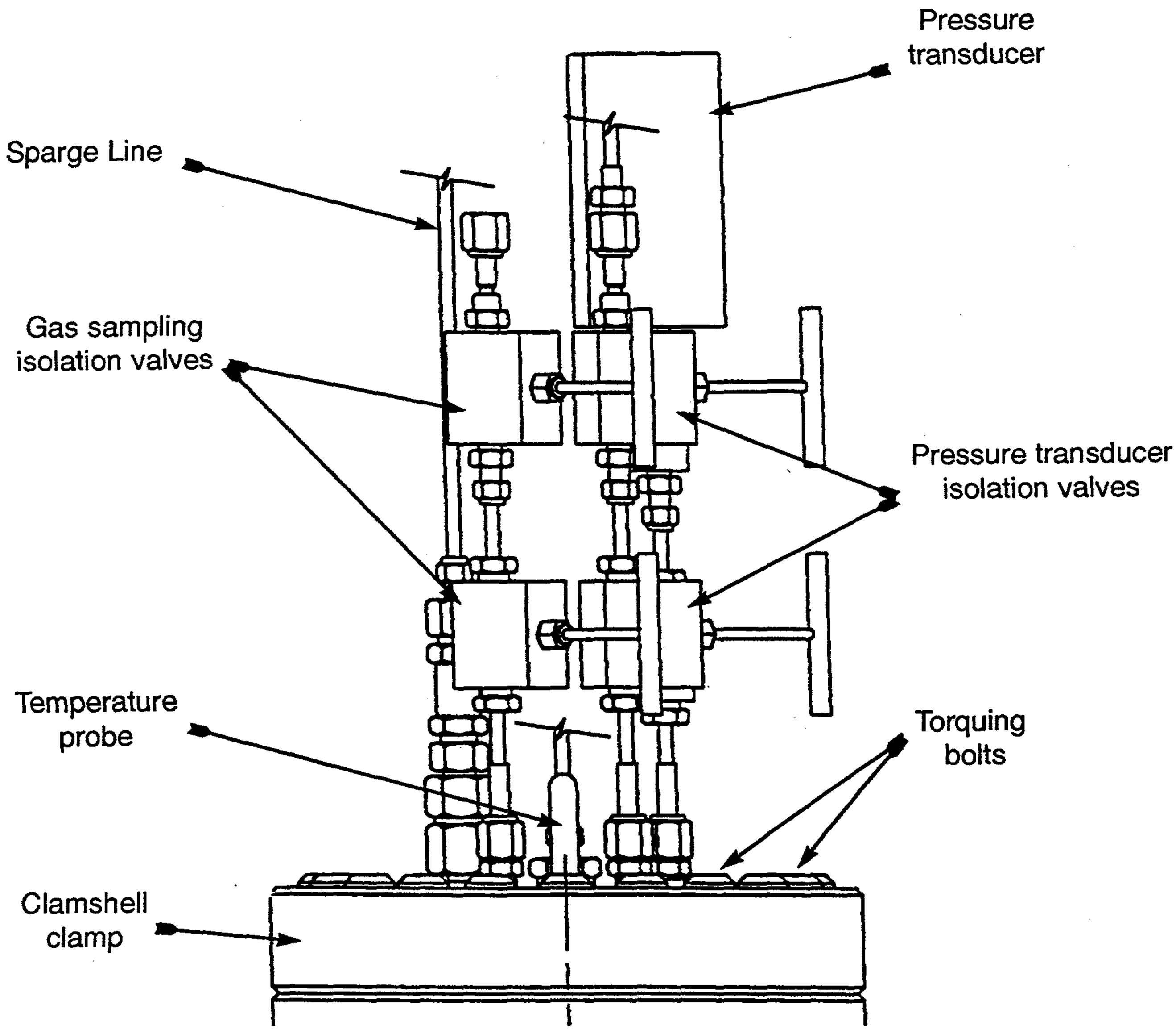
CONCLUSION

The art of helium leak testing with an MSLD is not an exact science. It is, however, a method of nondestructive testing that can be adapted to various situations with the use of a little imagination. When leak testing a contaminated system, it is important to pay close attention to minor details. With construction of the test system filters must be in place to ensure personnel safety and to protect valuable equipment from radioactive contaminates. Prior consulting and closely working with radiation safety personnel during planning and construction will save time and money. Following each test it is important to have filters and equipment monitored for radioactive contamination. This will preclude the spread of contamination if equipment is contaminated during leak testing.

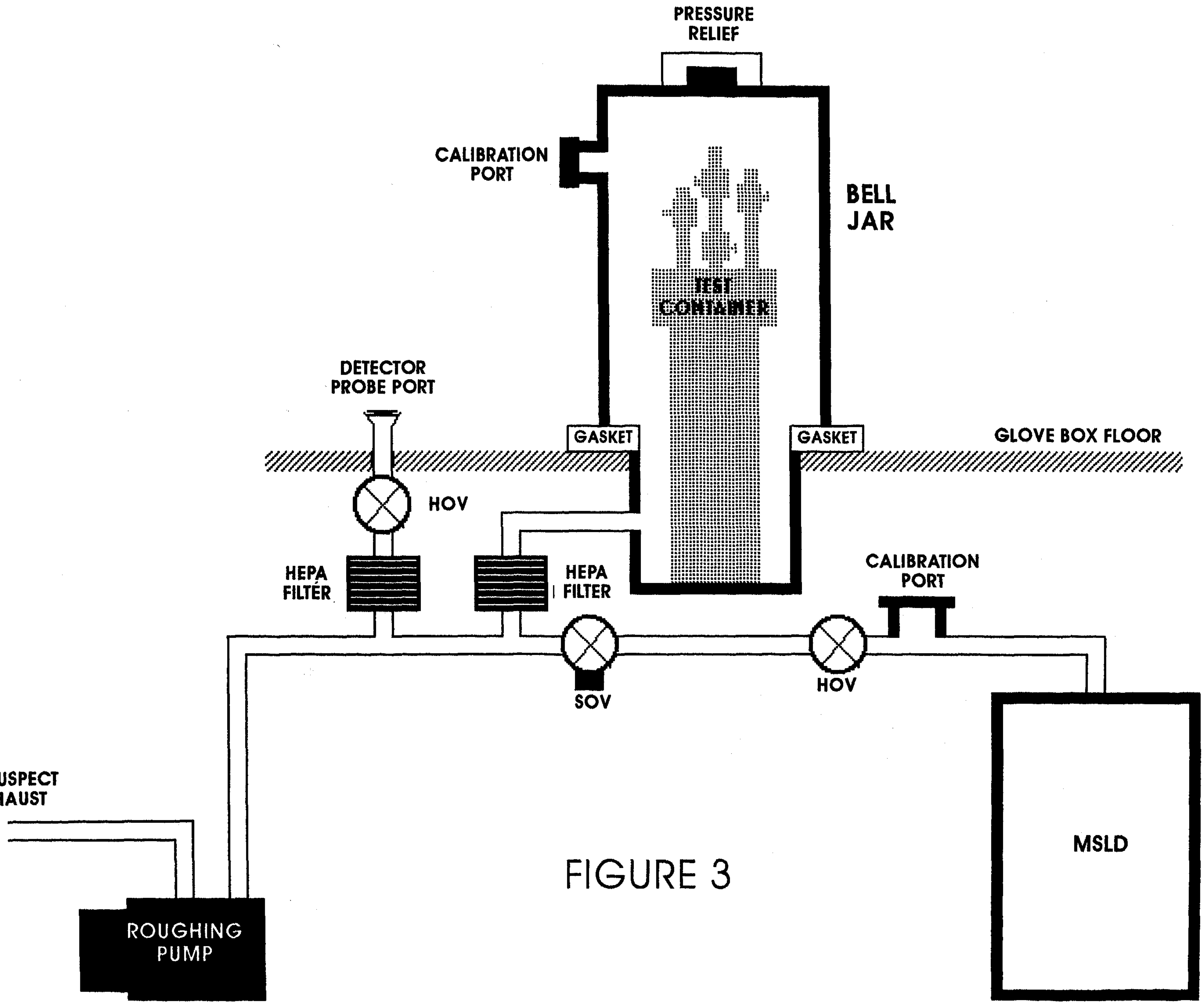
During early testing some of the minor details were overlooked and valuable time was lost. The use of a cover gas around the bell jar to minimize background levels would never have been tried if the test criteria had been evaluated prior to testing. Early leak-test problems with the container lid would have been minimized if the recommended lubricant would have been maintained on the torquing bolts. These are small details but cost large amounts of time.



GGE TEST CONTAINER
FIGURE 1



VALVE TREE
FIGURE 2


PRESSURE
RELIEF
CALIBRATION
PORT
BELL
JAR
TEST
CONTAINER
DETECTOR
PROBE PORT
GASKET
GASKET
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HOV
HEPA
FILTER
HEPA
FILTER
CALIBRATION
PORT
SOV
HOV
TO SUSPECT
EXHAUST
ROUGHING
PUMP
MSLD

FIGURE 3