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Helium Leak Testing Requirements in Fabrication of Cryogenic Storage Tanks

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ABSTRACT

Liquefied gases are used in a wide range of applications in many industrial sectors. The transportation of gases across the world is done in liquid state. Cryogenic liquids exist at extremely low temperatures (≤196 °C in many cases) which is why specialized equipment is required for their transportation and storage. It's vital for these tanks to be thermally insulated so as to maintain the low temperatures. The cryogenic stationary tanks are designed for long term storage of cryogenic liquefied gases under pressure. The maximum working pressure of cryogenic liquefied gas storage tanks ranges from 2 to 37 bar, depending on where the tank is to be used. The cryogenic storage tank can replace the reverse transportation of the gas cylinder, thereby saving a large amount of manpower and material resources to a large extent. Manufacturing of cryogenic tanks requires special technical and sophisticated fabrication techniques.

A non-destructive technique would be a significant aid in acceptance testing of new and refurbished tanks, saving significant time and money. Many welding processes are performed to construct cryogenic system. Leak-tightness for the cryogenic system is required at low temperature environment. Helium leak detection techniques are commonly used to find leak for the cryogenic system. The helium leak detection techniques for sniffing and pressurizing techniques are introduced. High vacuum is also necessary to use helium leak detector. Helium leak test is the best method for risk management in cryogenic system. Leak detection guarantees at test temperature. Leak-tight can be guaranteed at room temperature if leak is tested at room temperature.

In Inox India Limited, Kandla Helium leak detector employed to ensure leak tightness of the weld joints.

Keywords: Cryogenics, Leak testing, MSLT, Detection, Detector probe

1 Introduction

Cryogenics is a study of science which deals with the behavior of extreme low temperatures. Cryogenic liquids are extremely cold, with boiling points below -150°C. Such liquids include, liquid helium (-269°C), liquid hydrogen (-253°C) and liquid nitrogen (-196°C) - which is the most widely used cryogenic liquid. Liquefied gases are used in a wide range of applications, including metal processing, medical technology, electronics, water treatment, energy generation and the food industry. Today, more and more of these industrial gases are being delivered to customers in liquid form at cryogenic temperatures, enabling them to be stored on site for later use. Because of their low temperatures, the storage of cryogenic fluids is difficult. Cryogenic fluids must be maintained at low temperatures and high pressures, otherwise the change of phase may occur. It's vital for these tanks to be thermally insulated so as to maintain the low temperatures. Liquid Nitrogen is used as precoolant in most of the cryogenic systems. In steel industries, Oxygen is used in the production of steel. Basic Oxygen Furnace (BOF) uses oxygen instead of air. The use of inert gases in welding industry has initiated higher demand for gas production in the recent past. Nitrogen and argon are primarily used to provide an inert atmosphere in chemical, metallurgical and welding industries. Nitrogen is also used as a liquid to provide refrigeration, food preservation, blood, cells preservation, high temperature super conductivity. Argon is used in Argon – oxygen decarburization (AOD) process in stainless steel industry. It offers inert atmosphere for welding stainless steel, aluminum, titanium etc. The property of inertness of argon is used to purge moulds in casting industry. Cryogens like LOX, LH2 are used in rocket propulsion while LH2 is being considered for automobile. As more and more industries require the gases mentioned above, demand for cryogenic tanks keeps growing and is projected to grow at a higher rate than 5% CAGR (Compound Annual Growth Rate) between 2020 and 2026 [1].

Helium leak testing is a sensitive, reliable and most widely used method of leak detection in manufacturing industries. Helium leak testing with sniffer technique is being widely used in detecting leaks in today's fabrication industries. In this technique, the specimen to be tested is filled with helium or a mixture of helium and air to a pressure greater than atmosphere. The surface of the test object is then scanned with a "sniffer" connected to the leak detector. Helium flowing out through any opening will be sucked into the leak detector system by the sniffer and the leak rate is indicated [2]. It will be great to reduce risk before operate cryogenic system.

1.1 Components of Cryogenic Storage Tanks

Cryogenic tanks have three basic components: Inner Tank, Outer Vessel (Jacket) and Insulation. Inner and outer vessels are considered to be of cylindrical shape with hemispherical heads. Main components of Cryogenic Storage tanks are shown in Fig.1.

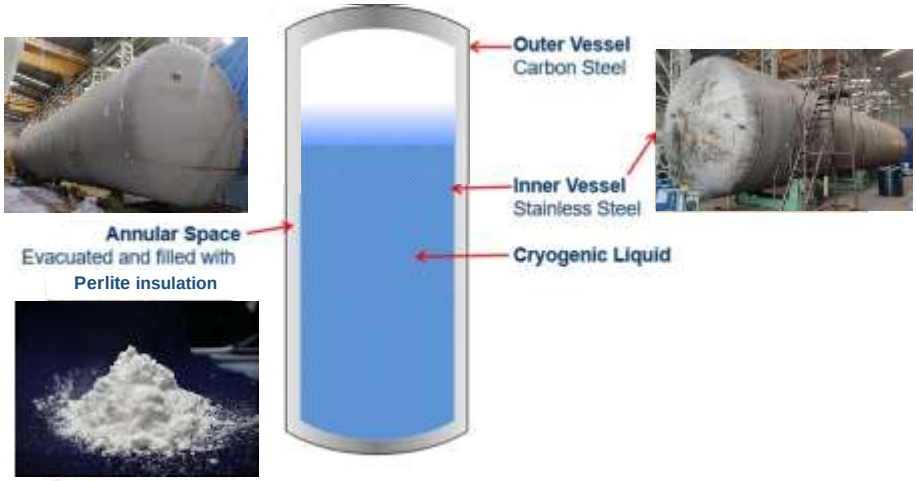

Fig.1 Main components of Cryogenic Storage tanks

The inner vessels and piping are made of stainless steel to ensure high-grade cleanliness – particularly important for the food and electronics industry. Austenitic steels, stainless steels, fine grain double normalized and tempered nickel steels, copper and aluminum are excellent materials, which can withstand cryogenic temperature. The material with Face Centered Cubic (FCC) or Hexagonal Closed Pack lattice have more slip planes. The slip planes assist in plastic deformation (rather than breaking) and hence increase the impact strength even at low temperatures. Austenitic stainless steels are suited for cryogenic applications as they remain tough and ductile even at -269°C. Stainless steel is the selected material for the inner vessel due to its higher strength to width ratio at low temperatures [3]. Stainless steel is the preferred material for manufacture of storage tanks, since it is easy to produce leak-tight welds, easy to clean and the wide availability of standard sealing systems.

Fig.2 Inner Vessel

An outer vessel made of carbon steel maintains the insulation and vacuum around the inner tank under normal operating conditions. Carbon steel is chosen as a material for the outer vessel because of its better strength at atmospheric conditions compared to low temperatures. The outside shell is specially coated (with anti-corrosion primer and a special environmentally friendly top coat) and a vacuum-perlite system with a molecular sieve adsorbent is applied to ensure outstanding insulation. The Outer Vessel has to withstand only atmospheric pressure acting on it. It would not fail because of excessive stress, but it would fail from the standpoint of elastic instability (collapsing or buckling). The inner tank holds the low-temperature commodity, while the outer shell is exposed to the ambient temperature. Typically, the outer container is not exposed to cryogenic temperatures.

Fig.3 Horizontal and Vertical construction of Cryogenic Storage tanks

These tanks can be in vertical or horizontal arrangement and they can be used for LIN, LOX, LAR, LNG, CO_2, LH2, LHe and N_2O according to customer requirement. Fig.3 shows typical horizontal and vertical construction of storage tanks.

Nozzles are connections through which the vessel is connected to the piping instrumentation and other control equipment. These are welded to the shell. Nozzles can be from seamless pipes, forged hollow bars. These are connected by means of flanges, screw type connections or directly welded.

All piping connected to the inner vessel must be suitable for cryogenic liquid service at -196°C. Due to very low temperatures, cryogenic pipes face unique corrosion and deterioration challenges. Annular space piping penetrating through the outer vessel must have an adequate thermal barrier. This thermal barrier prevents the carbon steel outer vessel from reaching embrittlement temperature. Annular space piping welds are not required to be passivated.

Different insulating materials can be used: from cellular glass, to rockwool and expanded perlite. Perlite is the most typical insulating powder used to separate the inner and outer shells of cryogenic tanks. It is light-weight granular material which is highly crushable when subjected to pressure [4]. Perlite is a fine granular inert material with excellent insulation properties. The distinguishing feature which sets perlite apart from other volcanic glasses is that when heated to a suitable point in its softening range, it expands from four to twenty times its original volume [5]. This expansion is due to the presence of combined water in the crude perlite rock. When quickly heated to above 870°C, the crude rock pops in a manner similar to popcorn as the combined water vaporizes and creates countless tiny bubbles in the heat softened glassy particles. It is these tiny glass-sealed bubbles which account for the excellent insulating properties and light weight of expanded perlite [5]. The space between the inner tank and the outer container is filled with perlite, which has the ability to maintain cryogenic temperature, and is subjected to high vacuum treatment. The air between the inner vessel & outer vessel are sucked out with the help of air separation unit and it is made vacuumed for better insulation [6]. An adsorbent is also added to maintain the vacuum in the insulation interspace. Perlite is then installed inside the tank using a pneumatic pumping system. Vibrating equipment is used to ensure correct settlement within the annulus and nozzle spacing. The vacuum and insulating material help reduce heat transfer, thereby reducing boiling of liquid stored in the container. Perlite minimizes radiative energy transfer between the two tanks [7]. The effectiveness of the storage vessel depends on the insulation hence it is very important parameter that should be considered in the design of the cryogenic storage vessels. Perlite insulation exhibits low thermal conductivity throughout a range of densities. The normal recommended density range is 40 to 80 kg/m^3 [8]. In addition to its excellent thermal properties, perlite insulation does not shrink, swell, warp or slump. It is easy to handle & install, noncombustible and relatively low cost. Because it is an organic material, perlite is rot and vermin proof [8]. As a result of its closed cell structure, the material does not retain moisture. Portable perlite expansion plants often are used to insulate very large storage tanks. In this, perlite ore is expanded on-site and the expanded perlite insulation is conveyed pneumatically into the annulus. For large liquid oxygen (LOX) tanks, the annular space is often kept at ambient pressure with a nitrogen purge to keep the insulation material dry [9]. Being a powder, perlite will settle over time, leading to the danger of transferring any loads from the inner shell to the outer shell. This can cause deformation of the outer shell, leading to damaged internal fittings [7]. Both the vessels are being designed using the assumption that the perlite offers negligible structural resistance, thus permitting the vessel to freely expand or contract under thermal cycles [4]. The perlite can work quite well up to a pressure of 2E^{-2} torr.

Fig.4 Air Vaporizer

Cryogenic vessels are equipped with ambient air vaporizers that convert the liquids to a gaseous state before they enter the pipeline. Ambient air vaporizers require no external source of energy; and enables vaporization through exchange of heat with the surrounding air. Fig.4 shows typical construction of air vaporizer.

Different gauges and valves are required to monitor and stabilize the cryogenic vessel and these gauges have a precise measuring capability, otherwise smallest change in the pressure variations can leads to trouble. Hence different parameters of the vessel are continuously monitored. All these gauges and valves must have the capability to work even at the higher pressure environments without any malfunction [6].

Skirt / Saddles provides support to the entire vessel. The skirt is usually welded directly to the vessel either to the bottom dished end or outside of the cylindrical shell. While saddles are welded with the outer shell in both the sides.

1.2 Design consideration in Cryogenic Storage tanks

Cryogenics vessels are designed for storage and transport of liquid gases at sub-zero temperatures [6]. The design approach from shape point of view is the cylinder with dished ends at both the ends. This design has good mechanical strength, larger volume ratio and is readily fabricated. The cryogenic storage vessel is designed in such a way that it has two different shells, first one is called the inner shell or product container and the other is called as outer shell and it is also known as vaccum jacket. There are many different design concepts for cryogenic storage vessels. However, most are of double wall construction with the annulus filled with expanded perlite. Super Cold or extremely cold cryogenic fluids such as hydrogen and helium are normally stored in spherical, double-walled vessels with perlite-filled annular spaces [7].

Cryogenic Storage tank where the temperature is maintained using a vacuum insulated tank and liquid nitrogen. The air between the gaps are sucked out with the help of air separation unit and it is made vaccumed. Thus, Cryogenic storage tanks are specially designed to minimize heat transfer and maintain very low temperatures. The tanks range in capacity from 3,000 to > 100,000 litres and come with standardised working pressures of 18, 22, or 36 bar respectively. The tank must sustain the working pressure otherwise the tank explodes and causes damage to itself and to its surrounding equipments [6].

At lower temperatures, the internal energy of atom is low. As a result, the atoms vibrate less vigorously with less thermal agitation [10]. The material becomes hard and brittle at low temperature. Properties of materials change, when cooled to cryogenic temperatures. Cold embrittlement (and failure) of materials may occur when inappropriate materials are subjected to extreme cold. At cryogenic temperatures, materials such as rubber, plastic and carbon steel can become so brittle that very little stress can break the material [11]. To avoid problems with cold embrittlement, materials like stainless steel, copper, brass, and most alloys of aluminum should be used in the cryogen containment systems.

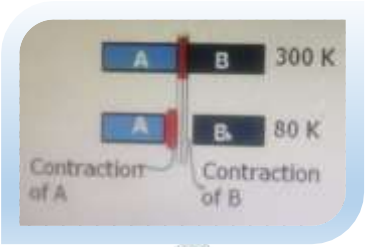

Fig.5 Reduction in the dimensions of a metals occur when cooled to low temperature

Reduction (contraction) in the dimensions (Shrinkage) of a metals occur when cooled to low temperatures as shown in Fig.5. The Coefficient of thermal expansion decreases with the decrease in temperature. Most of materials such as stainless steel make most of thermal contraction from 300 K to 77 K, so extra thermal contraction is very small from 77 K to 4 K [12].

Cryogenic gases significantly expand upon warming. Liquid nitrogen expands to the gas form at a rate of 1:700 [11]. Rapid expansion may create extreme pressure build up in an Outer vessel, which may result in vessel rupture or failure. Inner vessel must be permitted to expand or contract without damaging inner vessel, outer jacket, support system or annular space piping [13]. Hence, a knowledge of behavior and properties like strength, ductility, thermal and electrical conductivities etc. of material is necessary for the proper design. Inner vessels are made of austenitic stainless steel and cold stretched for selected medium / high pressure models. If the vessel is cold-stretched, the maximum design temperature cannot exceed 50°C.

Liquid densities are also being considered while parameters are determined. Different Cryogenic Liquids have different densities. They are, LOX : 1142 kg/m^3, LIN : 808 kg/m^3, LAR : 1393 kg/m^3 [13]. Boiling points of Cryogenic Liquids is also very important parameter while designing cryogenic storage tanks. LOX, LIN & LAR has Boiling points 90.18°C, 77.36°C & 87.3°C respectively [15].

Design Pressure of Storage tank : The inner vessel is designed to withstand the internal pressure and bending forces [3].
The Internal pressure : The internal MAWP (at the top of the tank) + Full vacuum in the annular space + Maximum liquid static head. Horizontal inner vessel have a minimum MAWP of 3.45 bar(g) [13].
The External pressure : Outer vessel's pressure relief device set point + Perlite compaction pressure + 0 bar(g) internal pressure in the inner vessel.

Purity requirements of the inner tank 9% Ni. Outer vessel is made up of Carbon Steel. And CS is highly susceptible to corrosion and minor defects may grow into leaks [14]. For Inner & Outer vessel, corrosion allowance of 0 mm is allowed [13].

The support components between the inner vessel and outer jacket should be designed such that they are suitable for cryogenic service as well as thermal expansion and contraction. The bottom of the skirt of the Vessel must be securely anchored to the concrete foundation by means of anchor bolts embedded in the concrete to prevent overhauling from the bending moments induced by wind and seismic loads.

1.3 Cryogenic Vessel Manufacturing

Typical Cryogenic Vessel Manufacturing Flow chart shown in Fig.6.

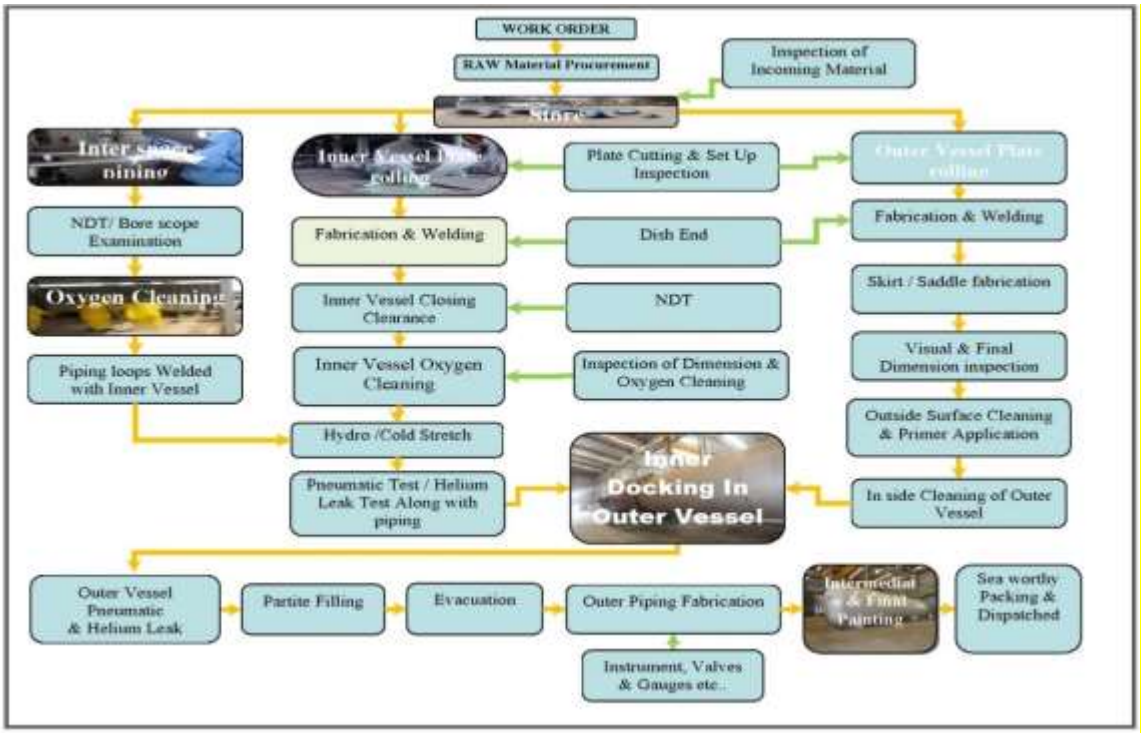

Fig.6 Cryogenic Vessel Manufacturing Flow chart

When rolling steel plate for inner vessel fabrication, care shall be taken to ensure that contaminants and residue on the rollers is not impregnated in the inner vessel material. It is recommended that independent and segregated rollers be used for stainless steel, otherwise the rollers shall be cleaned before use [15]. The key processes involved in the fabrication of Cryogenic storage tank is shown in Fig.7.

Fig.7 Key processes in Cryogenic Storage tanks manufacturing

Although cryogenic stationary tanks are based on a standard design, they can be customized as per specific application and requirements. The storage tanks are designed to meet individual customer needs and complies latest PESO regulations. Cryogenic Storage tanks may comply with the European Pressure Equipment Directive (PED) PD 5500, ASME Sec.VIII Div.1/2, AD Merkblatter, IS 2825 or LITS tanks (Leading International Tank Standard). The basic aims of the codes are same, but their approach is different.

1.4 Cold stretch test

To make wall thin and weight light, cold stretching technology is applied in cryogenic pressure vessel manufacture [16]. Cold stretching is carried out after all forming and welded steps are complete. Cold stretching (or Cold Working) increases the yield limit of a stainless steel material by work hardening. A Storage vessel is being Cold stretched by pressurizing it over 1.5 to 1.6 times the design pressure under careful control of the deformation Vs Pressure as outlined in ASME Sec. VIII Div.1 Appendix 44 [17].

Austenitic stainless steel (ASS) exhibits considerable work-hardening upon deformation while retaining the characteristics of the material. The high rate of austenite deformation hardening was utilized by cold stretching (CS) of cryogenic pressure vessels. A few percent deformation will give the vessel a considerable and homogeneous yield strength improvement, and the wall thickness may be greatly reduced [18].

Stresses in a Vessel made of SA-240 Type 304 Stainless steel is 138 MPa for cryogenic applications. After Cold stretching, the allowable stress is increased to 270 MPa (Table 44-1-1 ASME Sec. VIII Div.1) [17]. The resulting permanent plastic deformation increases the diameter upto 7% and the volume by 14% [19]. The pressure shall be increased until Pc (Cold Stretching Pressure) is reached. Pc shall be maintained until the calculated strain rate has dropped to ≤ 0.1 %/ hr. The minimum holding time ≥ 1 hr. The calculated strain rate shall be determined by repeated or continuous measurements of the circumference while the vessel is under Pc. The required maximum strain rate of 0.1%/hr shall be met during the last half hour.

2.0 Leak Testing

2.1 Leak, Leakage, Leak Rate, Leak Types

Leak is the unintended passage of a substance (such as gas or liquid) from a contained space to the external environment. It is used as a term to describe a flaw or failure in a sealed system or component.

It has become conventional to use the term "leak" to refer to an actual discontinuity or passage through which a fluid flows or permeates. "Leakage" refers to the fluid that has flowed through a leak. In other words, Leakage is measured by how much fluid passes through a leak under given set of conditions. "Leak rate" refers to the rate of fluid flow per unit of time under a given set of conditions, and is properly expressed in units of mass per unit of time. Because leakage will vary with conditions, it is necessary to state both the leak rate and the prevailing conditions in order to define a leak properly. Therefore, leak rate is often expressed as the product of some measure of pressure and volume per unit of time [2]. The leak-tightness is specified as leak rate which is the quantity of fluid leaking across the boundary per unit time [20]. Standard leak rate refers to the rate of flow of atmospheric air under conditions in which inlet pressure is 0.1 MPa ± 5%; outlet pressure is less than 1 kPa; temperature is 298 K ± 5 and dew point is less than – 298 K [2]. Leak types vary based on the nature of the defect. There are three types of leak, i) Gross Leak: This type of leak is usually large and is often visible to the naked eye or easily detectable using basic methods. It results from noticeable defects like large holes, unsealed joints, or broken seals. ii) Fine Leak: These leaks are smaller and harder to detect. They can result from microscopic holes, fine cracks, or minute gaps in seals. Their detection often requires more sensitive methods or instruments, such as helium leak detection or pressure decay tests. iii) Permeation Leak: This isn't a "leak" in the traditional sense, where a hole or gap exists. Instead, it's when a gas or vapor permeates through the walls of a container or system due to the molecular nature of the material itself. Materials like certain plastics or elastomers might allow specific gasses to pass through them at the molecular level, even if there is no physical breach. Common leak types include: Holes, Weak Seals, Cracks, Other Imperfections.

2.2 Purpose of Leak testing

- Leak testing is used to identify and quantify these leaks to ensure the integrity and safety of the system or product.
- Leak testing is carried out to check that a storage vessel does not have significant leaks at the design operating conditions.
- Leak testing is employed to detect leaks and determine the rate at which a liquid or gas will penetrate from inside a "tight" component or assembly to the outside, or vice versa, as a result of pressure differential between the two regions, or of permeation of somewhat extended barrier [2].
- Leak testing is performed for two distinct, but related, purposes: to locate the leak and to determine the leak rate [21].
- Leak Testing is generally the term used to describe low pressure testing with air or gas just to see if the joints in a piece of equipment e.g. flanges and threaded connections are leak tight after assembly.

2.3 Leak Testing Benefits

Leak testing offers numerous benefits from a non-destructive testing perspective. They include:

1. **Enhanced Product Reliability**: Leak testing ensures the integrity of components and systems, reducing the risk of failure or malfunction.
2. **Improved Safety:** By detecting leaks early, leak testing helps prevent hazardous situations and potential harm to personnel or the environment.
3. **Compliance with Regulations**: Meeting leak test pressure limits and adhering to industry standards ensures compliance with regulatory requirements.
4. **Cost Savings:** Early detection of leaks minimises costly repairs, product recalls, and potential liabilities associated with faulty products.
5. **Quality Assurance:** Leak testing identifies defects and discrepancies, maintaining product quality and customer satisfaction.
6. **Process Optimisation:** Integration of leak testing equipment enhances production efficiency and streamlines quality control processes.
7. **Environmental Protection:** Preventing leaks minimises the release of harmful substances into the environment, supporting sustainability initiatives.
8. **Data-driven Decision Making:** Leak testing equipment provides valuable data for analysis and continuous improvement efforts.
9. **Risk Mitigation**: Leak testing helps in understanding certain risks and threats, accepting that leak exist, and taking the appropriate measures to reduce their effects in case they happen.

2.4 Selection of system

Understanding the specific defect is crucial in selecting the appropriate Leak Testing Method. The correct choice of a leak testing method optimizes sensitivity, cost, and reliability of the test. One approach is to rank the various methods according to test system sensitivity. The various testing methods must be individually examined to determine their suitability for the particular system being tested. Only then can the appropriate method be chosen [22]. There are many methods of detecting leaks. Depending on the sensitivity – leak tightness required, suitable leak testing method to be selected [2]. A basic non-destructive test for leaks is visual examination [23].

The pressure test is performed either with a pressurized liquid (hydro test) often water, or with a pressurized gas (pneumatic test) often air. Hydro testing is the most common method of pressure testing and involves the application of pressure using water. Most leak testing methods involve pressurizing the equipment, although the pressure used may be well below the design pressure. Hydro test is a proof test which verifies structural integrity of the component under stress. If a pressure leak test is used and if the source of pressure is higher than the test pressure, the vessel should be provided with protection against overpressure by the use of suitable reducing valves, pressure gauges and pressure relief valves. In general, pneumatic leak testing should not be performed until the integrity of the vessel has been established by pressure testing.

If the job is to be leak-tight only up to gross leak range, that is, up to 10A std cc/s, only gross leak test should be performed. However, if the job is to be leak-tight in fine leak range, that is, less than 10A std cc/s, then helium leak test is recommended. If the job is tested by pressure-vacuum technique of helium leak detection the job shall also be tested by gross-leak test method to ascertain the presence of helium inside the job [20].

2.5 Leak location methods

The main methods used for leak location are: (1) pressure testing; (2) search gas methods. There are a number of leak location methods which involve pressurizing the vessel and then detecting leaks visually. The equipment may be pressurized hydraulically and liquid leaks observed. Detection of leaks may be assisted by the use of some kind of coating which shows them up more clearly. Alternatively, pressurization may be pneumatic and detection effected using a soap bubble or similar technique. If the vessel is sufficiently small to be immersed in a liquid, the evolution of air bubbles may be used to detect leaks in a manner similar to that used in repairing a bicycle inner tube puncture. Another group of methods is based on the use of a search gas. The gas is introduced into the air pressurizing the vessel and is detected by a suitable device [21].

2.6 Hydro Testing Vs Leak Testing

In order to ensure the quality of a retaining product, whether it holds a liquid or a gas, such products must have a certain level of leak tightness. While a 100% leak-tight vessel/product is not possible, you can always reduce leaks to the lowest possible level to mitigate any disastrous leaks. In order to assess whether a product is safe to use without fear of leakage, it need to be ensure that leak tightness can be quantified and proved [23].

Pressure testing is normally specified to check for leaks or determine whether or not there may be gross errors in design or fabrication that could cause the component to fail (fracture, crack or deform) under pressure. Pressure tests in shops are usually conducted with water (hydro testing) or with air (pneumatic testing) or a combination of both (hydro-pneumatic testing). As the name indicates, pressure testing involves testing with elevated pressures, often above that at which the component will normally operate, so safety is of utmost importance when witnessing a pressure test. Pressure tests must be conducted in accordance with the construction code or standard to which the item was built e.g. ASME BPVC Section VIII for vessels or ASME B31.3 for process piping. These codes generally indicate how to witness such a test safely after the pressure has equalized and stabilized. Whether testing by hydro test, hydro-pneumatic or pneumatic, the pressure testing equipment should have the means to prevent over pressuring the equipment under test.

Hydrostatic testing (Hydro test) and leak testing are both methods used to assess the integrity of pressurised systems, but they differ in their approach and purpose. The pressure test challenges the strength of the joint as well as its leak tightness. On one hand it is more demanding than the sensitive leak test because it tries to pry open the joint or defect under large pressure; on the other hand, it cannot detect the minute leak paths that a sensitive leak test can detect. Therefore, it is better not to try to label the pressure test (hydrostatic or pneumatic) as better or worse than a sensitive leak test; they each have a different purpose [24].

Table 1 Hydrostatic Testing Vs Leak Testing

	Hydrostatic Testing (Hydro test)	**Leak Testing**
Method	A hydro test involves pressurising a system or component with a liquid, typically water, to a predetermined pressure level.	Leak testing involves pressurising a system or component with gas or liquid and monitoring for the presence of leaks.
Purpose	It is primarily used to check for structural integrity and evaluate the strength of the component or system. It ensures that the item can withstand the expected operating pressure without permanent deformation.	The primary purpose of leak testing is to identify and quantify leaks or unintended fluid or gas escapes in a system or component. It ensures that the system or product is free from leaks that could compromise safety, quality, or performance.

The bubble leak testing method would be much less sensitive at 1×10^{-02} std cm^3/sec. and the hydro test leak sensitivity would be even less at 1×10^{-01} std cm^3/sec. Even a carefully prepared hydro test can be insensitive to leakage due to trapped air. Depending on your atmospheric conditions, the leakage water might be evaporating as fast as it's leaking, which also makes it tough to detect. Regardless of service, we always back up a hydro with an air leakage test using shop air at minimum, in case the hydro missed a leak. In fact, we usually use shop air at 5-15 psig to find the major leakage. The rule of thumb that people used is that under ideal conditions hydro reaches 10^{-2}, air underwater or bubble test can reach 10^{-4}, and He 10^{-6}. One way to look at this is to compare the viscosity of the test fluids, water, air, and He.

The sensitive leak test searches for minute leak paths, and can detect very low leak rates through tight pinholes, cracks, or joints. A sensitive leak test is typically conducted under a low differential pressure of the order of 1 atm differential (15 psid), and it uses sensitive detection techniques such as bubble solution, gas spectrometer, or acoustic or conductivity probe.

Leak tests are usually done at low pressures which are substantially below equipment design pressures to minimize risk of injury [25]. Leak Testing is not a one-size-fits-all process; its applications vary across industries. The common leak testing techniques are described in ASME V Article 10, and they consist of pressure tests and sensitive leak tests.

3.0 Helium Leak Testing

3.1 Why use helium for leak testing?

Helium is monomolecular and is the best atom for leak test. The reason behind it is as below,

- **Inert :** Helium atom has only one shell (K shell) which can have maximum 2 electrons. Thus its shell is already complete called duplet. It can neither lose electrons nor gain electrons. Hence, its valency is 'Zero'. Helium is an inert gas and does not react with other gases and materials in the system and does not corrode or damage metallic materials.

- **Light Mass** : Helium, having a low atomic weight (only 4 unified atomic mass units (u)), let it flow through small leaks more readily as compared to heavier gases [2].
- **Smallest Particle** : Helium is one of the smallest gas molecules that exists, allowing us to detect leaks down to 10^{-11} mbar L/s [26]. A helium is such a small molecule it can readily leak or permeate into the system unless strict precautions are taken to prevent leaks [27] & thus causing little interference in sensitive leak detection work. It's smallest size making it possible for them to escape through the smallest of holes – this makes it the perfect tracer gas for leak testing. Helium can flow well through narrow channels due to small collision cross section and non-interacting property.
- **Higher Velocity** : At any specific temperature, helium molecules have higher particle velocities than those of any other gases so that helium penetrates through leaks more rapidly than most other tracer gases. Thus Helium has a high mobility, so it diffuses quickly.
- **Negligible presence in atmosphere :** Air contains 5 ppm of helium (partial pressure of 5×10^{-6} mbar). It found only in small quantities (0.0004%) in the atmosphere.
- **Versatile & safe :** Helium is an incredibly versatile element. It is nontoxic, nonflammable and non-hazardous and therefore environmentally safe. Being inert, helium is relatively safe to use (rather than hydrogen) which makes it highly suited to many applications.
- **Relatively inexpensive :** Helium is relatively inexpensive.

The quantity of helium may become significant when testing large volumes, at higher pressures and/or at high speed. There are a number of techniques to reduce the consumption of the helium gas. One of these is to mix the helium with another lower cost gas, either nitrogen or compressed air. This is only possible where the sensitivity of the test is not compromised by the mixing process. Helium Gas shall be used having purity at least 99%.

3.2 Principle of Helium Leak Testing

Gas molecules entering the mass spectrometer are bombarded by electrons emitted from a heated filament. The ion beam produced by the electron bombardment is accelerated in the form of a narrow beam by means of an electric field. The ions then pass between the pole pieces of a permanent magnet. The magnetic field deflects the ions in circular path. The radius of the curvature of the path depends upon the mass of the ion. Ions having equal mass will all emerge from the magnetic field at a certain position. All light gases such a hydrogen will follow a smaller curvature, Only the selected gas (helium) will reach the target plate through the different "lenses" and consequently reach the pre-amplifier. The helium leak detector is adjusted so that only helium ions are collected. The flow of helium ions to the collector induces a minute electrical current which can be detected, amplified, and used to activate an electrical meter to control the pitch of an audio signal generator [2].

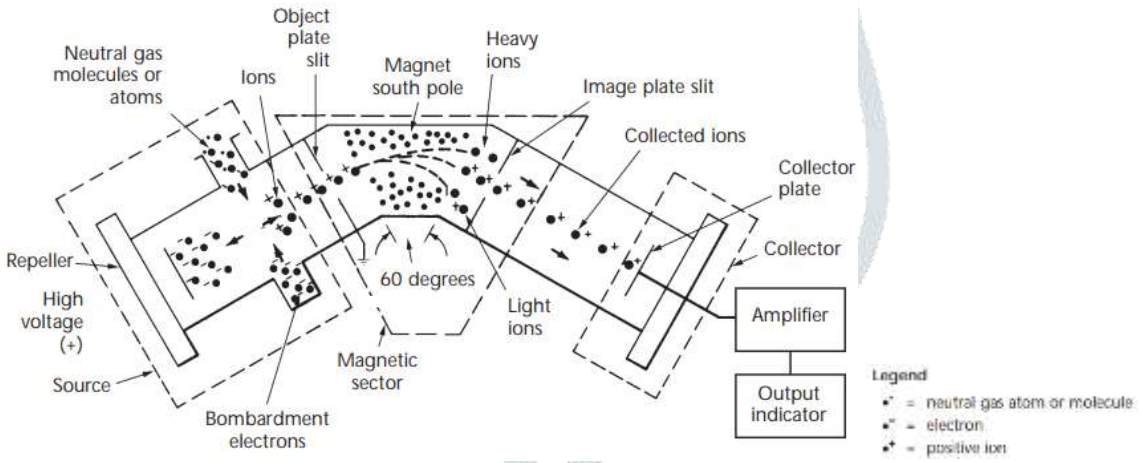

Fig.8 Helium Leak Testing Principle [28]

In brief, ionizing a gas containing helium, pushing it through a magnetic field and collecting the helium ion as they emerge.

3.3 Leak Detection

3.3.1 Factors for the Selection of Helium Leak Testing Technique

Following factors to be considered while Selection of Helium Leak Testing Technique

- Size, Shape and location of the equipment to be tested.
- Choice between use of pressure or vacuum or both.
- Maximum leakage rate specified or that can be tolerated.
- Degree of automatic leak testing operation required.
- Number of parts or complexity of the system to be tested.

3.3.2 Leak Detection Methods

There are four common methods of using a helium mass spectrometer leak detector to locate and measure leaks, namely (i) Detector Probe (Inside-Out), (ii) Tracer Probe (Outside-In), (iii) Hood Technique (Outside-In), (iv) Bell Jar method (Inside-Out). However, detector probe method is used primarily for inner vessel & tracer probe method for checking outer vessel.

3.3.2.1 Detector Probe Method : The test piece is pressurized with helium and a detector probe is passed over the outside surface to locate the leak. The mass spectrometer leak detector is connected to the hose of a scanning probe that collects samples of gas leaking from the external surface into the surrounding atmosphere. Helium Sniffing is used where the part can be pressurised above atmospheric

and a helium sniffer gun is manually positioned round the part. Sniffing test detects the escaping helium gas through a long distance sniffer probe from the part which the tested body is pressurized with helium gas. Fig.9 shows the schematic of sniffing test. The sniffer probe can be moved over potential leak areas and the leak can be located while the leak detector is not supposed to move due to turbo molecular pump in leak detector. When the leak is small, we can measure the total leak by collecting all the escaping helium gas with vinyl wrapping from all the potential leak area for about 5 minutes. Insert the sniffer into the vinyl wrapping to test leak after collecting the escaping helium gas for 5 minutes. The leak sensitivity is increased by collecting helium gas. It is better to reduce the collecting volume in order to increases sensitivity. Reference for sniffing test is air around the test area. Background reference is 5 ppm helium in air. There is no leak with sniffing test if leak detection is not observed. Ventilation is important to reduce background helium in air for sniffing test. When helium gas is released around the test area, the sensitivity of sniffing test becomes very low because the helium concentration of surrounding air is increased by the released helium gas [12].

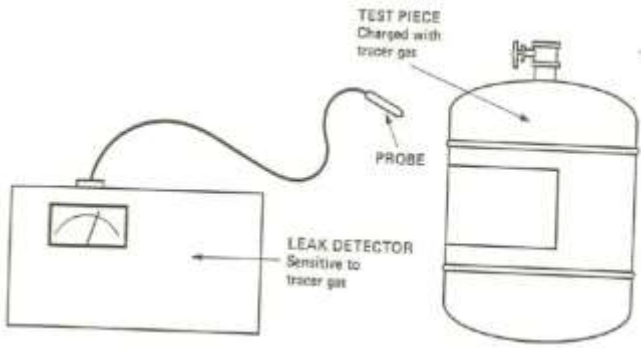

Fig.9 Detector Probe Method

Reference for sniffing test is air around the test area. Background reference is 5 ppm helium in air. There is no leak with sniffing test if leak detection is not observed. Ventilation is important to reduce background helium in air for sniffing test. When helium gas is released around the test area, the sensitivity of sniffing test becomes very low because the helium concentration of surrounding air is increased by the released helium gas.

3.3.2.2 Tracer Probe Method : Internal Pressure in combination of vacuum inside outer vessel is used for conducting a leak test. This method of testing is referred to as vacuum testing by tracer probe method. In this method, a fine jet of helium is passed over the exterior surface of the outer vessel. Due to vacuum, helium gas will be drawn into any opening through the welds or base metal and register on the leak detector as a visible or audible indication [2]. Test Pressure inside inner vessel : 4.2 kg/cm^2. Vacuum level between inner & outer vessel : 102 mTorr [29].

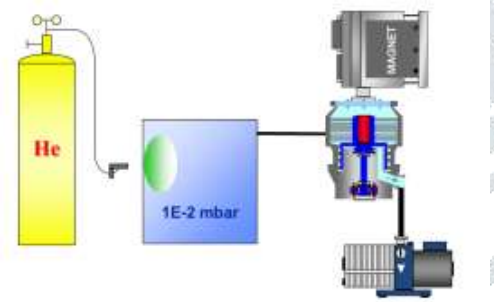

Fig.10 Tracer Probe Method

Detector probe & Tracer Probe both these both methods are qualitative rather than quantitative.

3.4 Equipment & Accessories

3.4.1 Helium leak detection Equipment / Leak Detector:

A device for detecting, locating, or measuring, or combination thereof, leakage. In most helium leak testing applications, a mass spectrometer is used to detect helium. A mass spectrometer is an instrument for separating or sorting atoms of different mass and which shall be capable of sensing and measuring minute traces of helium.

Leakage shall be indicated by one or more of the following signalling devices (i) Meter: A mbar liter/sec meter on or attached to the test instrument, (ii) Audio devices: A speaker or headphone that emits audible indications, (iii) Indicator light: A visible indicator light.

3.4.2 Search Units (Probes)

All areas to be examined shall be scanned for leaks using a detector probe (Sniffer) connected to the instrument through flexible tubing or hose. A sniffer is a device which sucks in traces of helium leaking from a job under helium pressure, when the job is scanned through the fine nozzle of the sniffer. Typically, it consists of a 15 cm long and 25 µm bore capillary tube, whose response is around 10s. The hose length should normally be restricted to about 2 to 3 m [20].

3.4.3 Pressure Gauges

Pressure gauge should be connected when carrying out pressure tests. At least 2 nos. calibrated Pressure gauge shall be used. Range shall be 1.5 to 4 times the intended test pressure [29]. The pressure-range indicated in the gauge should preferably be twice the intended maximum pressure, but in no case should the range be less than 1.5 times and more than 4 times that of the test pressure. All gauges shall be calibrated by suitable means and recalibrated at least once in six months. The gauge should face the operator controlling the pressure.

3.4.4 Vacuum meter

At least one calibrated vacuum meter to be attached to measure vacuum level of vessel. Vacuum pumps consist of rotary pump, diffusion pump, turbo molecular pump (TMP), ion pump, NEG pump, cryo-pump, etc. For instance, a leak detector consists of rotary pump and turbo molecular pump (TMP). The atmospheric pressure can be expressed as 1 atm, 760 torr, 1.01325 bar, 1.01325 × 105 Pa and 14.696 psi. Vacuum pressure can be considered as rough vacuum (RV) (1∼760 torr), medium vacuum (MV) (10^{-5}∼1 torr), high vacuum (HV) (10^{-8}∼10^{-5} torr) and ultrahigh vacuum (UHV) (＜10^{-8} torr). At least 2 nos. calibrated vacuum meter to be attached to measure vacuum level of vessel [29].

3.5 Sensitivity

As the speed of helium is three times higher than the speed of air, the amount of helium entering through a leak and thus the sensitivity is increased by a factor of 3 compared to air [30]. The smallest division on the meter, thus calibrated in std cc/s is the sensitivity of the equipment. Normally the working sensitivity of a leak-detector should be around 1 x 10^{-10} std cc/s for helium. The sniffer method, however, is a semi-quantitative method [16]. To measure small leaks, the system must be extremely leak-tight from atmospheric gas. Helium leak testing can generally be between one thousand and one million times more sensitive than using pressure decay techniques [31]. The high sensitivity of this leak detector makes possible the detection of helium gas flow from the lower pressure side of a very small opening of barrier separating two regions at different pressure or the determination of the presence of helium in any gaseous mixture. The detector probe is a semi quantitative technique used to detect and locate leaks and shall not be considered quantitative [29].

It is important to distinguish between the sensitivity associated with the instrument employed to measure leakage and the sensitivity of the test system followed using the instrument. The sensitivity of the instrument influences the sensitivity that can be attained in a specific test. The range of temperatures or pressures, and the types of fluids involved, influence both the choice of instrument and the test system [22]. The sensitivity of various test systems differ. If the test system utilizes a mass spectrometer operating in the detector-probe mode, the sensitivity of the test can be 10^2 to 10^4 smaller than that of the mass spectrometer itself [22].

The sensitivity of the instrument shall be minimum of 5 x 10^{-6} mbar liter/sec. If the sensitivity of instrument at any calibration is less than 5 x 10^{-6} mbar liter/sec, the instrument shall be returned, cleaned or repaired and recalibrated until the sensitivity can be obtained. The system sensitivity shall be determined before and after testing and at intervals of not more than 4 hours of testing [29].

The component to be tested shall, if possible be protected from drafts or located in an area where drafts will not reduce the required sensitivity of the test [29].

4.0 Helium Leak Testing of Cryogenic Storage tanks

Helium leak test is commonly used to construct cryogenic systems. Cryogenic system has many welding processes and leak-tight is required in the environment of superfluid helium at the temperature of 2 K [12]. The inner vessel, outer vessel and annular space piping must be Mass Spectrometer Leak Detector (MSLD) tested (e.g. helium leak tested) [12]. Equipment under Helium leak testing of inner vessel & outer vessel is as shown in Fig.11. The mass spectrometer leak detector is presently the most satisfactory and versatile means for performing rapid nondestructive leak tests with helium tracer gas in certain types of industries requiring minimal leakage rates.

Fig.11 Inner vessel & Outer vessel under Helium Leak Testing

Cryogenic containers have a vacuum chamber that guarantees the perfect isolation of the cryogenic liquids inside, which is why it is so vital to check for leaks [26]. Valves do not need to be leak checked.

4.1 Surface Condition : The surface to be tested shall be free of dirt, moisture, oil, grease, scale, paint or other contaminants that might mask a leak. If liquids are used to clean the component or if a hydrostatic or hydro pneumatic test is performed before leak testing, the component shall be dry before leak testing [29].

4.2 Surface Preparation : Surface preparation shall be done manually using wire brush. In case of any paint on external surface, it shall be removed by solvent cleaner. All openings shall be sealed using plugs or other suitable material, which can be readily and completely removed after completion of the test. Sealing material shall be tracer gas free [29].

4.3 Metal Temperature : The metal temperature during the test shall be maintained at 30˚F (17˚C) above the minimum design metal temperature, but need not exceed 120˚F (48˚C), to minimize the risk of brittle fracture. If the test temperature exceeds 120˚F (48˚C), it is recommended that inspection of vessel shall be delayed until the temperature is reduced to 120˚F (48˚C) or less [29]. The test pressure shall not be applied until the vessel and its contents are at about the same temperature.

4.4 Detection Time (Response Time) : The time required for an indication to appear and stabilize on the instrument output is the response time and it should be observed during system calibration [20]. The time required for a leak detector or leak testing system to yield a signal output equal to 63% of the maximum signal attained when tracer gas is applied continuously to the system under test. It is usually desirable to keep this time as short as possible [20]. To reduce the response time hose length shall be less than 15ft, unless the test set up is specifically designed to attain the reduced response and clean up time for longer tubing or hose length [20].

4.5 Calibration Leak Standard : To verify probe response before scanning the test object, the probe should be moved past the orifice of a known helium source at the same speed and distance as will be used for the test object.

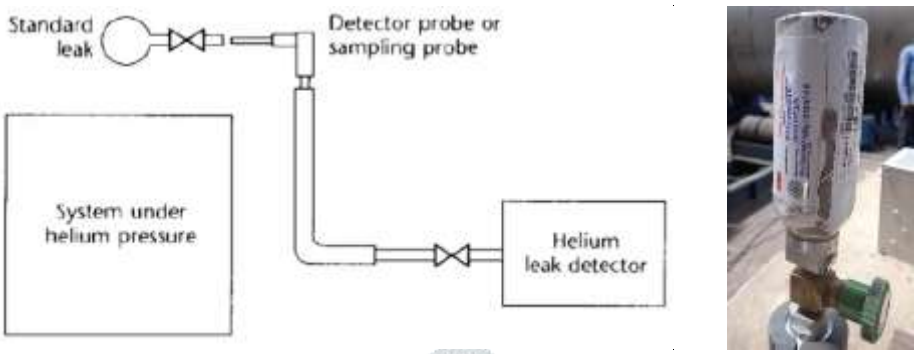

Fig.12 Standard leak calibration with Detector probe

Calibration leak standard shall be capillary or permeation type. The standard shall have at least leakage rate of 1 x 10^{-9} mbar liter/sec and maximum depletion rate per year is 2%.

4.6 Concentration of Tracer Gas

If the sniffer probe is placed in the line of the jet stream and at a distance not for away from the point of emanation, the probe would collect almost all of the helium that is leaked and at the rate at which is emanating. Then the detector probe technique can be said to measure the leak rate directly and fully. However, if the probe is far away or oriented at an angle to the normal, the measured value would be lesser than the true leak rate. At any location and orientation, the sniffer probe continues to measure the flux that is reaching. At a far distance and at oblique angles, the detector probe measurement is directly proportional to the helium concentration [32]. If required due to large volume, the tracer gas concentration could be 20 percent by volume. The concentration of the helium trace gas shall be at least 10% by volume at the test pressure.

Correction for concentration of tracer gas (helium) could be carried out by the relation:

$$\frac{MLR}{\%\ He} \times 100 = ALR$$

MLR = measured leak rate as seen on the meter, ALR = actual leak rate of component, and Percent He = Helium concentration [20].

4.6 Scanning Distance, Direction & Rate

4.6.1 For Detector Probe technique : Scanning Distance After required soaking time the detector probe tip shall be passed over the test surface. To reduce the instrument response and clean up time, probe shall be held on or not more than 3 mm from surface of device and moved not faster than 10 mm / sec [25]. The examination scan should commence in the lower most portion of the system being tested while progressively scanning upward. Start detector, warm up and adjust in accordance with manufacture's instruction before calibrating. Check for response of the probe, creating leak by mixture venting. Scanning shall be done sequentially for the area of interest and shall be identified the area examined and indicate point of leakage before start of further scanning. The scanning rate is determined by passing the sniffer probe connected to leak-detector across the orifice of the capillary leak standard, and should not exceed that with which 1 x 10^{-5} atm cc/s leakage from the calibration standard can be detected. Normally, a sniffer probe speed of around 25 cm/min is adequate. Increasing the probe scanning speed and test object-to-probe distance decrease test sensitivity.

4.6.2 For Tracer Probe technique : The tracer probe tip shall be kept within 1/4 in. (6 mm) of the test surface during scanning. If a shorter distance is used during system calibration, then that distance shall not be exceeded during the examination scanning. The examination scan should commence in the uppermost portion of the system being tested while progressively scanning downward [33].

4.7 Test Procedure And Technique : Hood / mask test is applied initially to check gross leakage. All areas to be scanned such as weld seam, Nozzles etc. are masked by plastic hood & sealed by adhesive tape. Now the areas marked during hood technique and balance weld joints as well as other areas are examined for leaks using detector probe connected to instrument through flexible tubing or a hose [29].

Unless otherwise specified, components which are to be pressure leak-tested should be pressurized to a minimum of 0.42 MPa (60 psi) or 15 percent maximum allowable design pressure, whichever is less, and held at this pressure for sufficient time till equilibrium is reached. The job is pressurized with helium. The device to be tested shall not be at a pressure exceeding 25% of the Design Pressure [ASME Sec V]. The test pressure has minimum soak time shall be held for 30 minutes [29]. Detector probe is pierced into the mask one by one and leakage checked. The areas showing the leak are marked for further testing. The component (Jacket space) to be tested shall be evacuated to a minimum vacuum level of 150 mtorr (2 x 10^{-1} mbar) and connected to MSLD.

4.8 Personnel Qualification Requirement

Test shall be performed by person certified as Level-I or Level-II in accordance with ASNT specification no. SNT-TC-1A. The test shall be evaluated by person certified as Level-II in accordance with ASNT-SNT-TC-1A Edition 2016 [29]. The persons carrying out leak-test should have undergone sufficient training in leak-test practices [20].

4.9 Acceptance / Rejection Standards
Leak size is a function of the size and shape of the opening (leak area) and the pressure. Leakage rate acceptance criteria shall be specified by the designer/purchaser.

4.9.1 For Inner Vessel with or without Interspace Piping (Under Pressure by Sniffer Method) : Unless otherwise specified by the referencing Code Section, the area tested is acceptable when no leakage is detected that exceeds the allowable rate of 1×10^{-4} std cm^3/s (1×10^{-5} Pa m^3/s)[33]. Considering the intended services of cryogenic vessel, the leakage rate shall not exceed 7 x 10^{-6} mbar liter/sec (6.93 x 10^{-6} std cc/sec or 7 x 10^{-7} Pa m^3/sec) [29].

Fig.13 Helium Leak Testing of Inner Vessel with or without Interspace Piping (Under Pressure by Sniffer Method)

4.9.2 For Outer vessel (Under Vacuum by Tracer Method) : Unless otherwise specified by the referencing Code Section, the area tested is acceptable when no leakage is detected that exceeds the allowable rate of 1×10^{-5} std cm^3/s (1×10^{-6} Pa m^3/s) [33]. Considering the intended services of cryogenic vessel, the leakage rate shall not exceed 5 x 10^{-8} mbar liter / sec. (4.95 x 10^{-8} std. cc/sec or 5 x 10^{-9} Pa m^3/sec.) which is having further restriction on acceptance criteria mentioned in ASME Code Sec. V [29].

Fig.14 Helium Leak Testing of Outer vessel (Under Vacuum by Tracer Method)

4.10 Examination of Repairs : When unacceptable leakage is detected, the location of leak(s) shall be marked. Depressurize the components. Repair the leak(s) as per referencing code. After repair has been made, the repaired area shall be retested as mentioned above.

4.11 Post Examination Cleaning : Generally, post examination cleaning is to be done by solvent cleaner [29].

4.12 Test Report : The test report shall contain, as a minimum, the following information as applicable to the method or technique: (a) date of test (b) certified level and name of operator (c) test procedure (number) and revision number (d) test method or technique (e) test results (f) component identification (g) test instrument, standard leak, and material identification (h) test conditions, test pressure, tracer gas, and gas concentration (i) gage(s) — manufacturer, model, range, and identification number (j) temperature measuring device(s) and identification number(s) (k) sketch showing method or technique setup [33].

5 Conclusion

Leak testing is considered a very important quality control tool and all joints and components of cryogenic storage tank are subjected to helium leak test using mass spectrometer leak detector (MSLD) at room temperature. Whether you need to avoid leakage under pressure or apply vacuum, the good thing is that a leak is not hard to identify if you have the right tools and the proper knowledge. Helium leak testing is the quickest NDE method for medium and large sized components. For most leak testing applications, the minimum leak size that an instrument can detect is unimportant since most users are interested in finding a leak and not necessarily measuring it with a high degree of precision. Higher the degree of leak-tightness required, higher is the cost of leak testing in terms of instrument, man power time, etc. With technological evolution, Helium Leak Testing will continue to play a pivotal role in ensuring the integrity of cryogenic vessels.

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