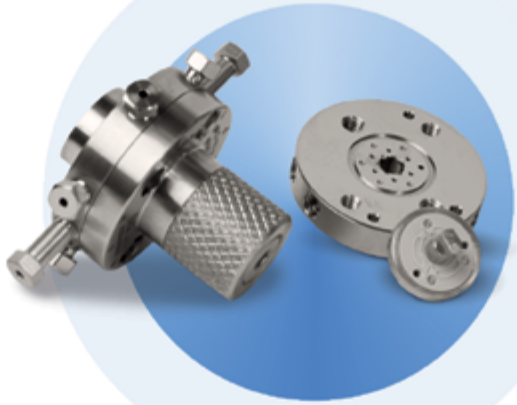
Technical note · 01

Leak Detection System and Method

The most sensitive technology for valve quality control



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Introduction

The following patented method is used to test all CVProducts valves during development and for quality control during manufacturing.

It could also be used to validate complete sampling systems capable of being highly sensitive or tuned for high leaking value. This portable system becomes a really valuable tool for valve and fitting manufacturers of all types. It is also a very convenient tool for process plant start-up teams, who need to validate sample or process lines for leaks. When used for this application, the argon purge flow also fills the need to evacuate air from the sampling system. After the leak test, lines could be capped to keep the atmospheric air out.

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It is mainly useful for a "capillary" type leak. Here we classify leaks into two large categories, i.e., capillary and orifice types. An orifice-type leak is defined as a fluid going into another area of a fluid system through a flow channel, where the length of the channel is similar to or less than its diameter, as shown in Figure 1A. Typically, an orifice leak could appear through the wall of a vessel or gas canister, tubing or fitting with a bad sealing area, etc. Small molecules go through such leaks very easily. It is then much easier to detect an orifice-type leak with a gas that has a low molecular size, like helium or even better H_2. However, due to its unstable nature, using H_2 as a tracer gas is unsafe. In resume, orifice type leak calls for small molecule gas.

A capillary-type leak is defined as a leak channel having a length much bigger than its diameter, as shown in Figure 1B. That kind of leak could appear in various valve designs, like globe, ball, butterfly, gate, etc., but also through fittings, generally because they are not properly tightened or because the surface of the tube is porous or scratched. A capillary-type leak is more easily detected with a tracer gas having low viscosity, i.e., with less resistance to flow. Generally speaking, the molecule size of any tracer gas is much smaller than any leak path diameter.

We have found that when using nitrogen to detect such a leak, we got about 4 times better sensitivity than a helium-based detector (see in Figure 7 a comparison between leak detection system sensitivity).

It is a general practice to use a helium-based mass spectrometer to measure leaks in valves, fittings and other systems in the industry. This is normally done by connecting the mass spectrometer to one side of the system, pulling the vacuum and checking for helium presence into the mass spectrometer. In such a system, the mass spectrometer is tuned to detect helium only, screening out atmospheric contaminants and other gases, from the measuring zone. The other side of the system is normally exposed to atmospheric air, where, generally speaking, there is a 5 PPM helium content.

Sensitivity could be increased by using a helium cylinder instead of relying on the helium content of atmospheric air. Such a leak detection system is calibrated by pumping atmospheric air or helium into the mass spec, through a calibrated leak orifice.

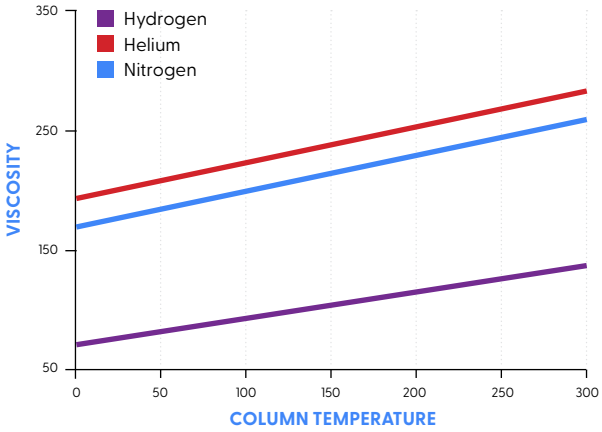
A helium tracer gas-based system is expensive. It needs a vacuum pump, a complete mass spectrometer, vacuum hardware, etc., and is often installed on a mobile cart; its cost ranges from $35,000 and higher.

Furthermore, for a leak detection system that uses a helium cylinder to increase sensitivity, the cost of helium becomes an issue. The actual helium supply shortage makes this situation even more difficult and costly. Recently many attempts to replace helium in analytical systems and other applications have been documented.

Table 1: Some tracer gas physical properties (at TPN)

	Molecule Diameter	Kinematic Viscosity
Nitrogen	4.0Å	1.51 E-5 m^2/s
Helium	2.56Å	11.40 E-5 m^2/s
Argon	3.8Å	1.34 E-5 m^2/s

Table 2: Chromatographic performance of Capillary Columns



Since our target is to measure capillary-type leaks, the use of N_2 as a tracer gas results in many benefits. One of them occurs when using an N_2 cylinder as a source of tracer gas. Industrial N_2 cylinders can be used in the system. Normally the price of these cylinders is set below $100. This feature makes our system much more affordable compared to a helium one.

Furthermore, atmospheric wair could even be used, since there is about 79% of N_2 in air, compared to 5 PPM for helium.

The difference in viscosity improves sensitivity. The N_2 viscosity is less than helium, so N_2 flows more easily in a capillary-type leaks (see Table 1).

Considering all the above-mentioned facts, we have designed a leak detection and measuring system based on N_2 instead of helium. This system is suitable for capillary and permeation type leaks, as we will demonstrate here.

Figure 2 shows one possible embodiment of the system.

System Description

Testing Valve

First, the Valve Under Test, i.e., VUT, is installed into the system. Here an ON/OFF valve of any type is shown, i.e., could be a diaphragm-based, ball, globe, etc. The size is not an important issue, since the purge argon flow is adjusted accordingly.

The inlet of the VUT is connected to a source of N_2 or air. Here, the source is pressurized to allow leak measurement at different operating pressures.

The VUT is in the OFF or CLOSED position. The outlet of the valve is then connected to a special tube arrangement that allows purging with argon the discharge side or volume of the VUT. The purge flow is then collected by T_2 and flown into the N_2 measuring system.

A step-by-step description of the operating sequence is shown below:

Step 1

When the VUT is installed and secured, V3 is open and V4 is switched to Port 2 for venting. This allows the evacuation of atmospheric air from the outlet section of the valve. This is done by a static purging or dilution purging to improve the removal of atmospheric air from the dead volume that VUT may have in its outlet zone. The V4 port 2 is switched alternatively between open and close position, resulting in pressure pulsing, diluting residual air. After a few repetitions, 5 to 10 for example, the system switches to Step 2.

Step 2

V4 Port 2 is closed and V4 Port 1 opened. V1 is switched on Port 2. In these valve positions, the argon purge gas coming through V3 goes through the VUT outlet zone, purging all its volume, evacuated through T2 tubing, going through V4, V1, Trap, $+H_2O$ and the PED before being vented through flow measuring device FT1.

Note: Here PED is a plasma emission detector tuned for N_2 measurement. It will also be possible to use other N_2 sensitive detectors, such as a micro Thermal Conductivity Detector (TCD) or even a mass spec. However, we do prefer the PED due to its sensitivity and the possibility to be more specific to N_2 impurity like the mass spec is for helium. Furthermore, such measuring devices could detect different wavelengths that are specific to other impurities, like O_2, and it is also possible to work in differential mode, compensating for baseline shifting.

The PED creates a luminous discharge into the flowing argon and a specific N_2 emission wavelength is measured. Different emission wavelengths could be used. Please see [REF 1], published by the author for more information about this technology.

Since the luminous discharge is generated by a low-power plasma, it is affected by moisture. To avoid any interference, a moisture trap 3A is installed. This trap is made of a molecular sieve having 3 Å of diameter to avoid trapping N_2 that has a molecular size of about 4 Å.

Next, some water is added to the argon carrier by the device $+H_2O$. A few PPM is enough, for example, 3 to 8 PPM. The combination of trap 3A and moisture doping device $+H_2O$ makes the PED insensitive to moisture pollution. A hydrocarbon trap is installed before the moisture trap.

The hydrocarbon trapping material could also be in the same tube as the moisture trap. The moisture could be added with the help of a permeation tube, or having one of the walls of the PED made permeable to H_2O, releasing moisture directly in the plasma zone.

Moisture pollution does not damage the PED, but slows down its response time, increases noise and reduces sensitivity.

Finally, the choice of the PED as a detector is also governed by the fact that this detector will not be damaged in any way by pollution, O_2, moisture or overload. It can also be turned OFF and ON without affecting the system calibration.

Step 3

The PED is measuring the N_2 present in the argon flowing into it. This value is sent to the signal processing sub-section of the system. The actual N_2 reading could partly come from residual N_2 purging out of VUT outlet zone, and carried by the argon purge. This would result in a decreasing signal as shown in Figure 3, where the actual N_2 value is going down by the purging action of the argon. Since the inlet N_2 pressure of VUT is still at atmospheric, no N_2 is flowing through VUT, so the leak rate is probably null or extremely low at the atmospheric pressure value.

Now system baseline is offset/re-zeroed to the current value, and then PC1 pressure controller raises the VUT inlet pressure measured by pressure transducer PT1 at predetermined step values. The value of those steps depends on VUT. For example, let's assume VUT is specified to have a working pressure of 1000 psig. The step values could be set by 100 psi increase. So PC1 will allow N_2 flow to raise the pressure at 100, 200, 300 and up to 1000 psig. There is a preset waiting time between step increments based on VUT. Larger valves will have a longer waiting time. During the N_2

inlet pressure stepping sequence, the PED measures N_2 signal and reports it to the signal processing subsection. If there is a diminution of the slope value of the signal S, N_2 is flowing through a leakage passage of VUT. Here the differential value could be measured to report the PPM of N_2 and transform it in the desired leak rate engineering unit. A band could be defined above the slope signal where the leak rate could be acceptable (see Figure 4).

Before each pressure increase, the system baseline is re-zeroed, so that monitoring is done within the same impurity range window.

A step change in S value could also be detected to report a leak. This is shown in Figure 5. The leak value offsets the signal of a constant value. Since the capillary channel area and length are constant, the leak flow will be constant, if the inlet pressure is constant. Furthermore, the leak flow rate will have a linear relationship regarding to the inlet pressure, since this system is in sonic mode, i.e., inlet pressure is much higher than outlet pressure. In this case, the slope value will still quite be the same. So measuring a step and/or slope change leads to leak detection. A very little leak will tend to change the slope without an appreciative step change of the signal S.

Step 4

When the VUT has been tested, the pressure regulator PC1 is closed and V6 is opened to depressurize the inlet side of the VUT.

At the same time, V1 is switched to Port 3 to allow pure argon to flow through the detection system, keeping it ready for another cycle. V3 is closed to stop argon purge flow through VUT.

Optionally here, the ports of V1 and V2 could be all closed, isolating the detection system. This way, the detector is still filled with clean argon, but there is no flow. The detector could then be shut down and kept like this until ready to test another valve. This also saves power in a battery-operated system.

Testing sampling system, see Figure 6

When testing a sampling system, an argon cylinder is installed to the sample point connection, to allow argon flow through the entire sample system. The analytical system detection (GC, mass spec, or other) is replaced by the leak detection system. When the sampling system has been properly purged with the argon, the leak detection system valve V4 allows this argon to flow through it. By having the argon purge flow at the low pressure and varying its flow value through the sampling system, the N_2 reading is monitored for leaks. Any atmospheric N_2 diffusing or leaking through the system will be detected. Normally a high sensitivity channel is used, like 337.1 or 357.69 nm. This model has shown much higher sensitivity than the helium mass spec leak detector.

2.1 A) Electronic control and signal conditioning subsystem

First, the signal from the PED is fed to the first stage of amplification, and fed to two independent zeroing sections (offset shifting), a range amplifier and A/D converter.

The first signal processing channel is used to display the PPM value of N_2 in the background or purge gas. It provides a direct reading of the level of leak through the VUT in terms of PPM of N_2. Other engineering units are generated through conversion done by the microcontroller unit.

The second signal processing channel allows setting the baseline at 50% of the scale. This way, it is not necessary to wait for a complete evacuation of N_2 out of the system. Up to 10% of N_2 can be offset by a 50% shift of the scale. However, after the initial purge cycle, the PPM value of N_2 will be well below 100 PPM. The system reads the actual value at the beginning of a cycle, records it, and subtracts it from subsequent reading.

This way, the gain of the system could be high, to provide PPB sensitivity, even if there is a high level of N_2 in the background.

Furthermore, the offset and gain control provides the possibility to detect and quantify low and high levels of leakage.

The electronic gain of the system could be changed to allow different systems full scales. However, this must be used in combination with the proper N_2 emission wavelength. Indeed, at low levels of N_2, PPB or PPM up to about one percent, the N_2 emission wavelengths of 337.1 and 357.69 nm could be used. The 337.1 nm provides slightly higher sensitivity while the 357.69 nm is much less subject to interference by hydrocarbon indeed, the plasma in the presence of hydrocarbons and N_2 will generate NH emissions at 336 nm. This could be an issue if VUT has a grease or oil trace. At a higher value of N_2, the emission wavelength of 406 nm will get better results and a more linear response.

To achieve this, different interference band pass optical filters are used to filter out the required wavelengths. On the other side of each interference filter, there is a photodiode connected to an operational amplifier. This constitutes one measuring channel. For example, three measuring channels could be differently configured.

Each one of the channels is connected to an analog multiplexer to allow individual channel selection as needed. The multiplexer is controlled by the microcontroller.

During the execution of a leak-finding procedure, the signal value may decrease and go under scale because the atmospheric or residual N_2 is being purged out by the action of argon flowing and purging the outlet side of VUT.

At this point, it becomes necessary to set back the signal on scale by offsetting it, at a predetermined value. For example, 50% of the scale, other values will work too. It may be desirable to change this value based on operating conditions, mainly the valve size, or the dead volume to be purged. These affect the rate or speed of the purging effect.

So, when the signal decreases below a certain value, the baseline line is automatically reset at 50% at the scale. This way the signal is always on scale to monitor any change caused by any leak.

2.1 B) User's interface

A graphic representation of the impurity reading provides a quick and efficient way to illustrate the performance of the system being tested. Furthermore, leak results could be saved on file for any components or systems being tested. Several leak system detections could be networked together and reporting to a remote-control system or PC.

2.2 System variation

a) Recycling

The system could be optionally equipped with a gas recycling system to decrease gas consumption. This is an advantage in a portable system where it may include a miniature high-pressure cylinder. In this case, the system could be kept in standby mode ready to use by having the gas circulating in a closed loop while keeping the detector ON.

The recycling system consists of a pump, a purifier and a flow restrictor F_R. The pump is used to increase the gas pressure, read by PT2, at the value required to maintain the desired flow through the orifice F_R, read by FT1.

F_R is a capillary flow orifice that is used to get flow through the system based on the pressure read by PT2, which is maintained at the desired value by controlling the pump by-pass valve V5.

PT3 is used to read the detector vent pressure when using the recycling system. This is an absolute pressure transducer. It is important to maintain detector operating pressure at a constant value. With this configuration, if the PED pressure tends to decrease due to pumping action, V5 will open to recirculate the gas. It may decrease the flow through the detector but this has little effect on the N_2 reading. Pressure variation will cause important baseline shift, so PT3 have the priority over PT2.

The purifier is trapping N_2, O_2, H_20 and hydrocarbons below an acceptable level. It could be made of a first bed of molecular sieve 13X, H_2O, in series with a second bed of Nickel or Copper catalyst for O_2 scavenging.

Finally, a bed of a getter base on zirconium alloy is used to eliminate or reduce the N_2 level. Other appropriate getters, catalysts or zeolites could be used as require.

b) Detection system and tracer gas

Despite the fact that the standard proposed configuration is very convenient, other PED and gas combinations are feasible if necessary. For example, it is possible to replace the argon purging gas with helium. The tracer gas could be argon instead of N_2. Any argon leaking through the valve will have a dramatic impact on the helium plasma or discharge. The argon will generate a strong emis sion line. This configuration has extreme sensitivity, so the PPT of argon could be detected. Furthermore, the above system operates the PED at atmospheric pressure, but operating it under vacuum conditions increases sensitivity and decreases baseline noise. This is also true for the standard configuration. This is achieved by entering a different set point for the PT3 pressure value.

Another possible testing sequence would include predetermined flow interruptions in the system, allowing tracer gas more time to diffuse within the system. When flow would be restarted, the carrier gas would bring within itself a higher concentration of tracer gas. This way, sensitivity could be increased and gas consumption would decrease.

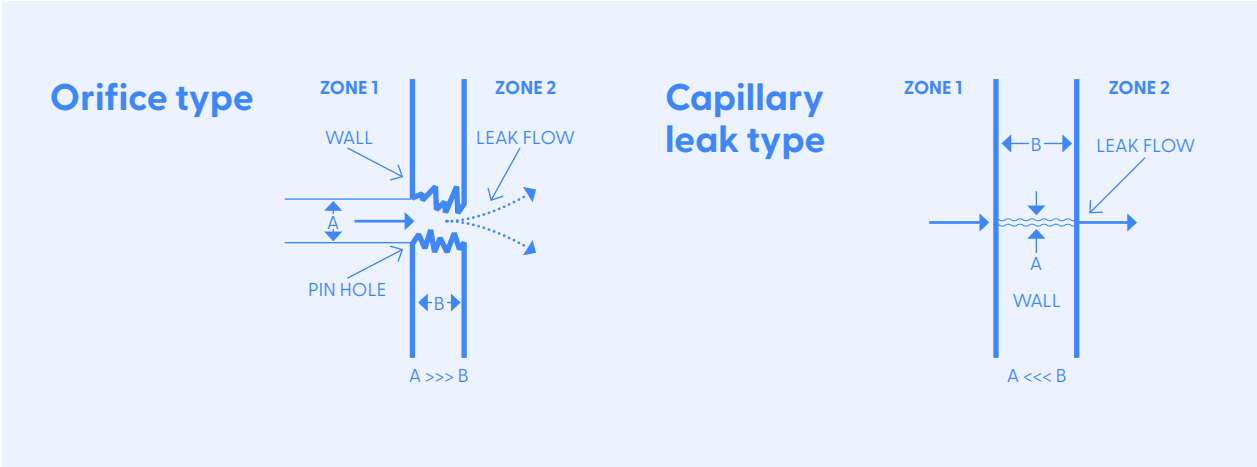


Figure 1 – Leak Types

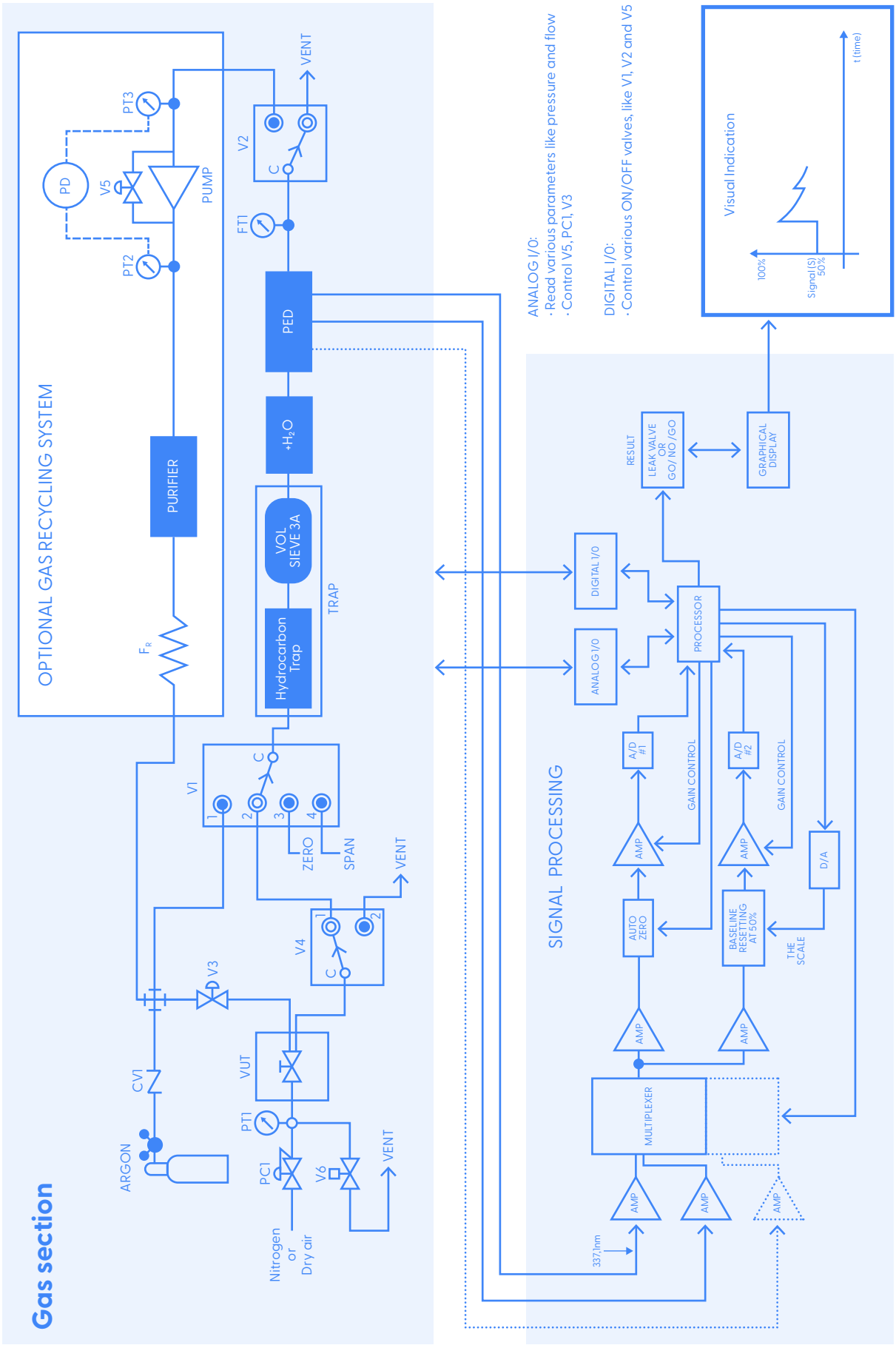


Figure 2 – Leak detection possible embodiment.

Bill of Material for Figure #2

PC1: High-pressure electronic pressure controller based on a proportional EDV-1.

PT1, PT2, PT3: Absolute pressure transducer

V1: Diaphragm type stream selection valve, CVProducts EDVS-4.

V2, V4: Diaphragm type 3-way switching valve CVProducts EDV-3.

V3: Tight shut-off proportional valve based on CVProducts EDV-1.

V5: Diaphragm-based proportional valve based on CVProducts EDV-1.

V6: Diaphragm-based ON/OFF valve, CVProducts EDV-1.

CV1: Check the valve to prevent back flow when using the recycling system.

P_F: Particle filter.

TRAP: Tube having proper gettering material for trapping H_2O and NMHC.

+H_2O: H_2O permeation tube before the PED or on one of the PED wall.

FT1: Electrical flow transducer

PUMP: Miniature reciprocating pump (KNF or other) with BLDC motor.

F_r: Capillary flow orifice.

PURIFIER: Custom made purifier.

Fittings: LipSeal fitting

Tubing: Coated Tip Tubing (CTT)

PED: Miniature plasma emission detector.

Electronic Sub-System: Electronic control Board and user interface designed.

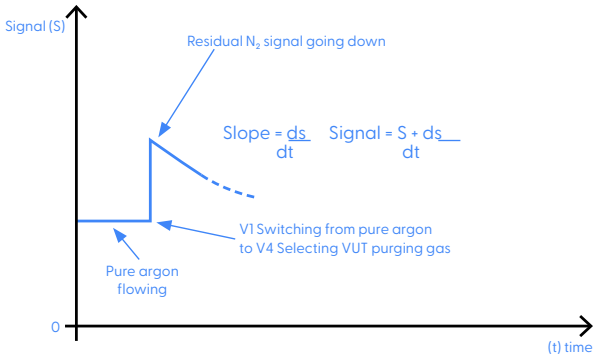


Figure 3 – Example of impurity signal

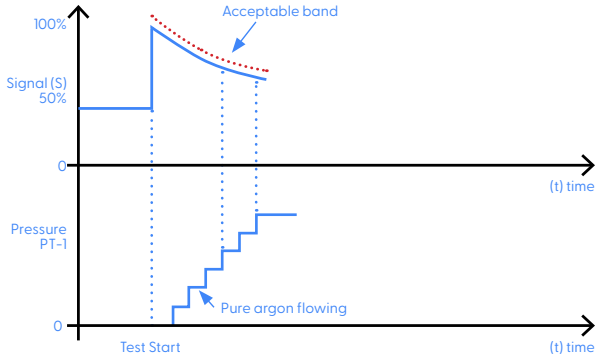


Figure 4 – Acceptable range band

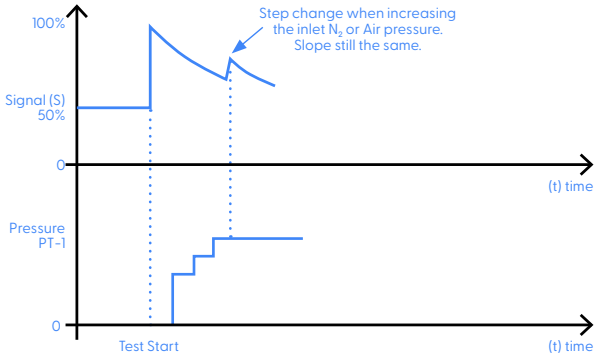


Figure 5 – Impact of pressure change on system signal

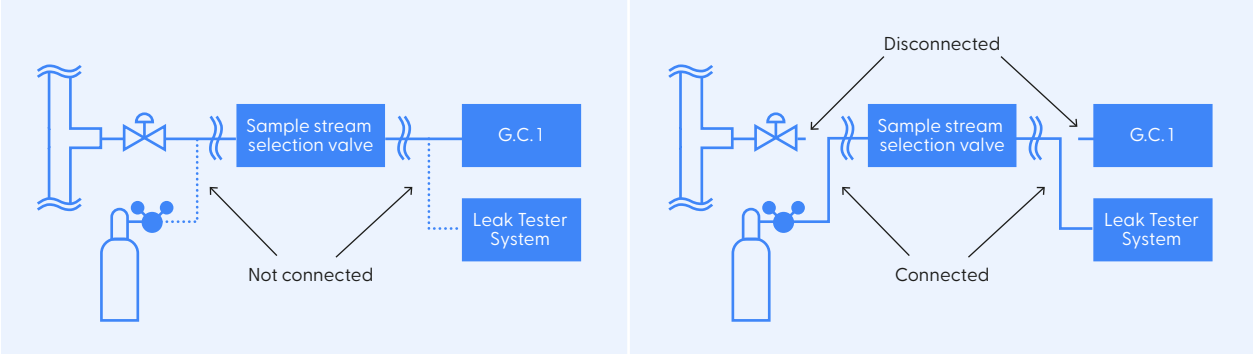
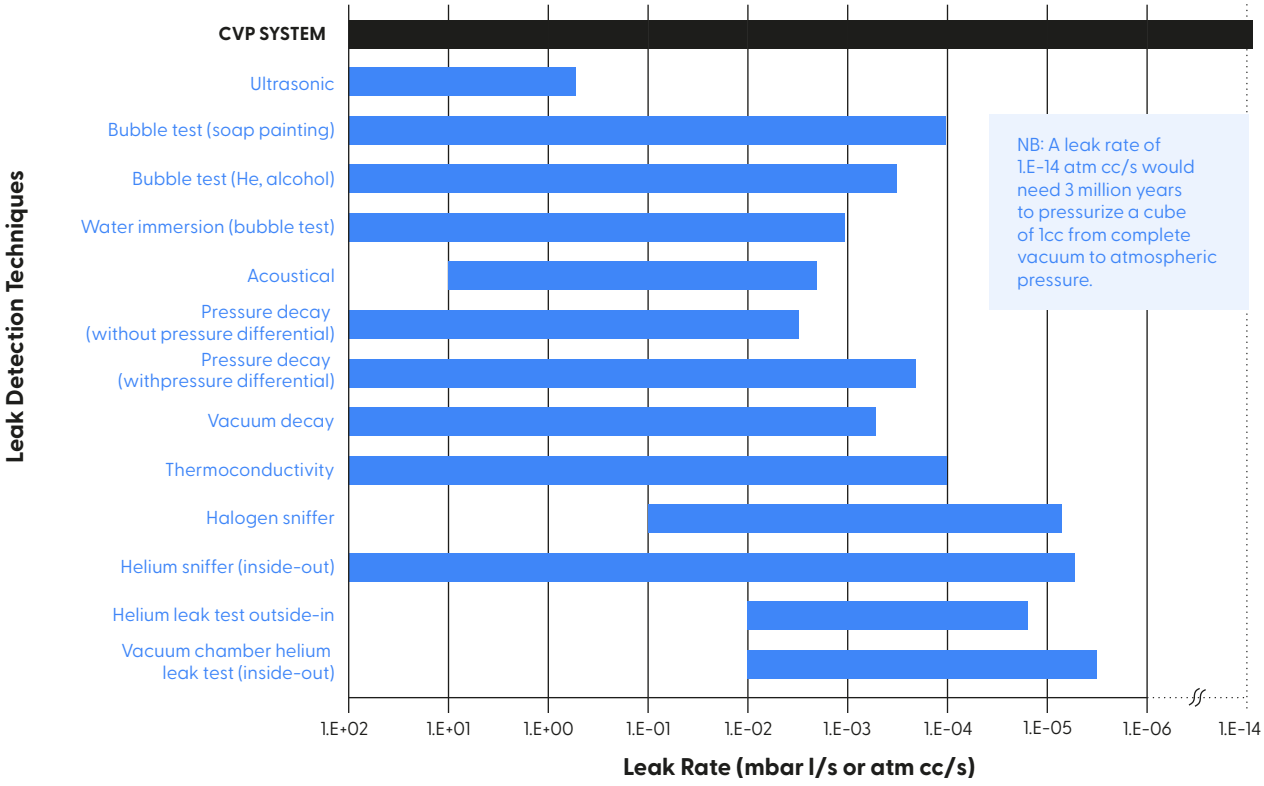


Figure 6 – Sampling system test schematic

Figure 7 – Leak Detection Sensitivity Comparison



References

[1] Gamache, Y., Lamontagne, A., and Langevin, M. (2023). AN-21 N_2Sense for interference-free measurement of N_2 in Argon and Helium. https://asdevices.com/wp-content/uploads/2023/05/AN-21-N_2Sense-for-interference-free-measurement-of-N_2-in-Argon-and-Helium.pdf.

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