NOTE TO READERS

The information contained in this application note may not be applicable to all processing operations utilizing the same or similar materials or analytical systems. Competent scientific and engineering assistance should be obtained when configuring the design, operation and maintenance of sampling and measuring instruments. It is the responsibility of the user to make sure that any particular system is safe for the operation. Compliance with approved standards in the area in which the system will be used is mandatory. Generally speaking, by law, the employers must protect their workers from being harmed by dangerous substances. This process starts during the design step.

Safety first !

1.0 INTRODUCTION

The scope of this application note is to guide ASDevices customers in the configuration of sampling systems and GC when working with reactive and/or toxic sample matrices. It also applies to reactive or toxic impurities in a stable or inert sample background. The first target is to create a safe working environment for people at proximity of the sampling system and the GC itself. The second target is to avoid equipment damages caused by a reactive sample leaking into the equipment. This application note should be used in conjunction with specific hardware manufacturer’s instruction used in designing such analytical system. All work should be carried out by trained and competent personnel.

All this, while having stable and repeatable measurement, ensures minimum system maintenance and long working lifetime. There are many different chemical processes in the industries which have their own characteristics. In electronic industries for example, several reactive and/or toxic gases are used in the process of fabrication of semiconductor electronics like operational amplifiers, microprocessors, photovoltaic devices, etc. In these processes HF, ClF3, silane, phosphine, ammonia, chlorine, boron trichloride, nitrogen trifluoride, fluorine, bromine and hydrogen are highly flammable, reactive, corrosive and/or toxic. Accidental leaks and fires of these gases are a major safety concern. Several of the most dangerous toxic gases like arsine, phosphine, silane play a critical role in the microelectronic industry. Refineries produce many hydrocarbons that are flammable and/or toxic. Dealing with a common compound like gasoline requires some special considerations for material selection. Therefore, it is very important that users recognize the inherent safety risks associated with the use of specific chemical compounds as they have the potential to not only damage equipment, but also to seriously injure operators of such equipment. The cost of safety is always too expensive before an accident occurs. Don't pay the price for lack of safety.

When dealing with such gases, the use of automated purged gas panels is mandatory. Furthermore, strict manipulation procedures must be rigorously followed. The list of chemical compounds entering in hazardous category is long, and beyond the scope of this paper. See reference 1 for some examples. In all cases and at some points, analytical measurement involving such nasty compounds must be done.

In some cases, the measurement is done directly into a challenging sample matrix and other ones consist of identifying reactive compounds as impurities in something more stable.

In both cases, great care must be taken to eliminate any leak out or into the system. The maximum concentration in air for silane is 5 ppm and 100 ppb for phosphine [1]. If higher than these values, there is a risk for the safety of lives. This imposes specific requirements on sampling and analytical systems.

While there are precise regulations, norms and references that exist and must be followed to manipulate such chemical compounds in process industries, paradoxically, those are only partially followed in the design of analytical systems or instruments. We have seen sample injection valves handling toxic products, installed inside a GC cabinet, without special procedures and specific hardware. We have seen OEM configuring process GC with improperly selected valve materials. Be careful about the self-proclaimed ''valve experts''.

We have also seen many different sampling systems and many dramatic results, and this right from the start. Generally, valves and detectors are components that will fail prematurely if some basic rules aren't followed.

In this paper, we will try to give general guidelines and common-sense practices. Each case has its own particularities. Good engineering practices must be followed. If you have any doubt, stop and consult. Don't take our recommendations for granted. Do your own verification to make sure your design is safe and follows any local regulations that may apply. Any design must comply with local code(s).

2.0 APPLICATION EXAMPLE

TYPICAL APPLICATION

Here, we will use a typical reactive gas sampling application in order to underline critical and practical details where special care must be followed. With the world consideration for environmentally friendly energy sources, we are experiencing an increasing demand for photovoltaic panels.

Photovoltaic panels convert solar energy into usable electricity. In the process of manufacturing such panels, silicon is required. There are chemical industries focusing on producing chemical compounds where the silicon will come from. Silane and chlorosilane are chemical compounds produced at large scale to fulfill this need.

These days, there is an increased demand for those photovoltaic devices and we have seen many system integrators and OEM involved in designing analytical systems using ASDevices hardware for such applications. If some critical and practical considerations aren't followed, even the best GC valves in the market will fail to perform and may put users and equipment at risk.

KNOWING THE SAMPLE AND THE RISK ASSOCIATED WITH IT

Silane and chlorosilane are reactive compounds and, as such, they are what we may call a challenging sample matrix. In our case, these chemical compounds will react instantaneously with the oxygen and moisture contained in the atmospheric air.

Such spontaneous reaction (Reference 2 shows interesting pictures of silane and chlorosilane spontaneous reaction) will lead to localised heat generation or heat spot. Generation of by-products such as HCl coming from reaction with moisture and SiO_2 from reaction with the atmospheric contaminant are a permanent risk. SiO_2 is generated in powder form and it is very abrasive. In fact, it is quartz powder. This powder will eventually accumulate into the sampling system and be carried away into sample valves. GC valve rotors will rapidly be scratched and internal leaking will occur between valve ports. The valve body will also become corroded by the HCl produced by the reaction of the sample with air leaking into the system. Finally, outboard leaking will occur and create a risk of fire and hazardous situations.

The same phenomenon will happen with a GC diaphragm valve. The diaphragm will gradually be punctured by the SiO2 powder allowing the sample to contaminate the actuator zone of the diaphragm valve and it will eventually result in a sample leaking into the surrounding atmospheric air. Once again, a high risk of fire and hazardous situations occurs. The higher is the sample pressure, the higher is the safety hazard in case of direct sample leak into the surrounding atmosphere. One could easily figure out the possible damage that such a leak could do to process GC. Even worse is the risk of personal injury.

Generally speaking, a rotary valve or a Purged Lip Sealing Valve (PLSV) will last longer than a diaphragm valve when operating under such conditions. The reason for this is relatively simple: there is more polymeric material compared to a diaphragm valve. Diaphragm thickness doesn't normally exceed .005 inch. In case of contamination by SiO_2, the diaphragm will get damaged faster. Nevertheless, any valve type not being used correctly will be heavily damaged, which may cause safety issues when the pressurized reactive fluid leaks out because the sample have been contaminated. The only difference with the rotary valve is the problems will be slightly delayed, but the end result will be the same. The PLSV will outperform both the diaphragm valve and rotary valve. The reason is the innovative purge principle. It is impossible for a leak to develop between two adjacent ports due to the purge channel. Any leak will be channelled into the purge and vent safely without causing the valve to fail for a much longer period of time compared to the rotary valve.

Many users will request the use of exotic alloys for the valve body. Despite the fact that some alloys are chemically compatible with the corrosive compound generated by chemical reaction with moisture, in this case HCl, the valve will be damaged by SiO_2 anyway. Indeed, the abrasion effect will damage the components made of Teflon or other compatible material. To a lesser extent, the valve body will also be eroded by SiO_2 powder. The valve will not seal properly anymore because of the scratches done by accumulation of SiO_2 into the system. The same applies for diaphragm valve, the diaphragm will be damaged. So even with specific alloys that resist to chemical attacks, the sample will leak into surrounding atmospheres and there is a high risk of fire and workers injuries. Figure 1 shows a damaged rotor, resulting in a major valve failure.

''The first step is to make sure that we fully understand the properties of various chemical compounds that our system will have to work with.''

Figure 1—Damaged rotor by erosion and chemical attack. Vespel rotor used with gasoline sample for a period of 6 months. Wrong material selection. Teflon or peek at ambient temperature would have been much better choice.

Special attention must be given to the overall system design. All possible sources of atmospheric contamination must be eliminated. All possible sources of hazardous samples leaking into the surrounding atmosphere must be eliminated or controlled. Here we are not only talking about direct leak but also surface contamination, diffusion and permeation.

Not only valves will be affected by contamination but also sample lines that become corroded and contaminated with particles. Depending on the phase of the sample i.e. liquid or gaseous, this situation is different, since fluid velocity and viscosity is different. The following figure shows typical damage done by chlorosilane contaminated by air. The valve and detector was removed from a process GC.

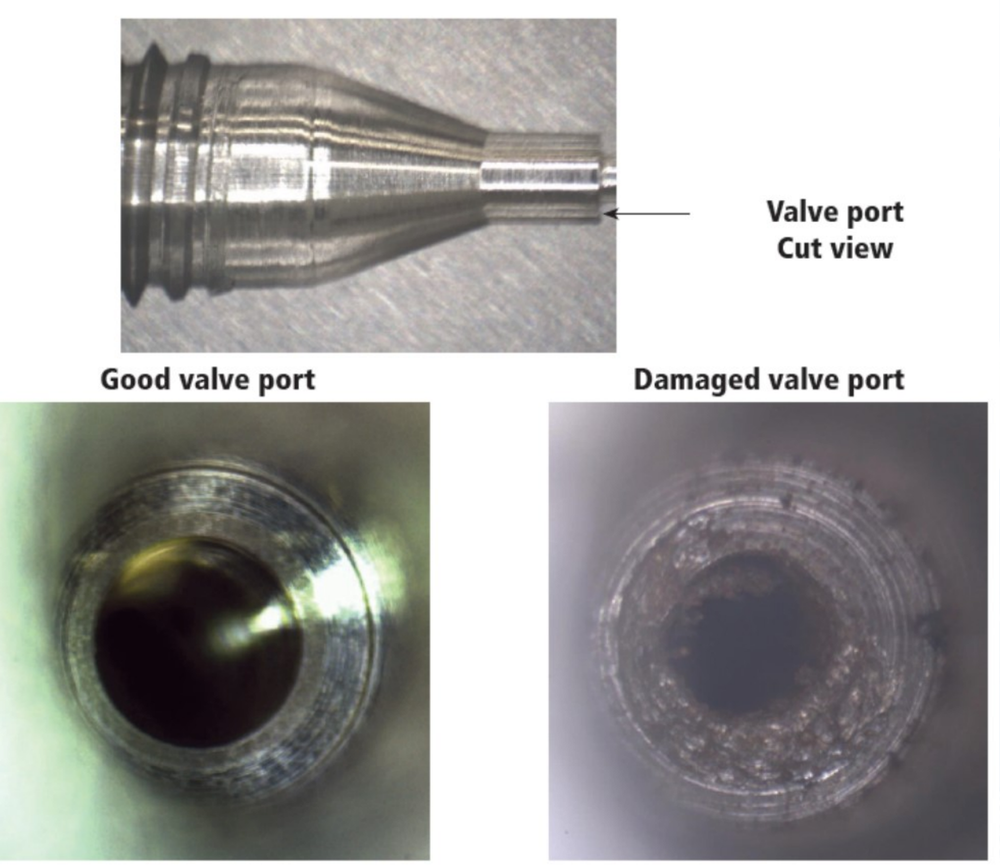


Figure 2A—Valve port damaged by sample contamination, the taper area is completely corroded, sealing ferrule is also affected. It's a dangerous situation, sample making slowly its way out of the system. A normal port picture is shown for comparison.

Figure 2B—Detector filled with SiO_2 (Top) and FID jet clogged by SiO_2 accumulation (Bottom).

Figure 2C—Results of chlorosilane mixture cylinder valve leak

Figure 2A shows a picture of a GC diaphragm valve damaged by a contaminated reactive sample. In this particular case, the sample is chlorosilane at 700 psi. Photo shows a port connecting the sample loop and the separation column. The diaphragm and the actuator were also damaged. This can result in hazardous situations, if proper care is not taken to evacuate leak with the help of an inert purge gas.

Figure 2B shows the accumulation of SiO_2 in a detector. Indeed the detector was clogged by SiO_2 which caused flow restriction and damages to the flow sensor device. The problem was traced to a contaminated carrier gas with 7 ppm of O_2. Here the detector is based on a plasma discharge in a quartz chamber. The ionized molecules trigger chemical reactions between O_2 in the carrier and the silane sample. In this specific GC configuration, the first component to fail was the detector.

The source of contamination was the electronic pressure controller (EPC). Indeed, atmospheric contaminant was diffusing into the carrier gas where the manifold mounted proportional valve was located. In this case, since the sample was pure silane, no corrosive by-products was generated.

Figure 2B also shows a clogged FID jet. The obstruction was caused by SiO_2. We have also seen some OEM having their TCD elements corroded. This is caused by having some sample reaching the detector because of a leaking valve when heart-cutting. However in all these cases, carrier or sample was contaminated. This is the real cause of a sequence of events that leads to system failure.

Figure 2C shows the result of chlorosilane mixture cylinder valve leak. The white deposit is SiO_2, generated by reaction with air. This can also happen to a GC valve, installed into an instrument.

It is obvious that special consideration must be followed when dealing with such sample. Not only the valve material and configuration must be properly selected, but all the system, starting from the sample point connection, up to the process GC must be designed and used in order to allow safe operation and get precise analytical measurement. We have seen many system integrators and OEM involved in designing analytical systems using ASDevices hardware for such applications. If some critical and practical considerations aren't followed, even the best GC valve in the market will fail to perform. When there is a valve failure in such application, the valve is not the source of the problem, but more a sequence of events that leads to sample contamination, and eventually causes the valve to fail. Obviously we presume that the valve has been properly selected, as initial condition.

3.0 DESIGNING A SYSTEM

Now that we know about the sample characteristics, we can start the system designing process.

BASIC PHILOSOPHY

Keeping in mind that we must be sure that any part of a fluid system coming in contact with the challenging sample should not react with it, we are going to use a passive and inert sampling system.

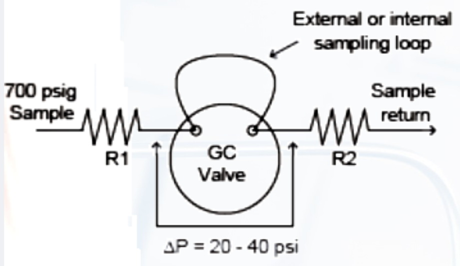
This includes the sampling lines, valves, filters, flow / pressure sensors and flow meters, etc. Every component outside the GC must be inert relatively to the sample being handled.

Inside the GC (process or laboratory), the same basic elementary rule applies. Every internal component in contact with reactive samples must be inert and prevent outboard leaks and contamination. By passive or inert, we mean that the nature of the fluid will not be chemically modified while in contact or flowing through such components. So what goes in will come out unchanged except for flow or pressure.

For this particular application i.e. silane and chlorosilane sample, we know what reacts with this sample, i.e. H_2O and O_2 from atmospheric contamination or from other sources like bad quality carrier gas or surface contamination. Permeation or diffusion through various seals, poorly designed sampling system and working procedures not adapted to the situation are also sources of contamination and problems.

CONTROLLING THE SAMPLE FLOW

Typically, the sample flow is set by the pressure drop between the properly selected sample and return points. In our example, the sample is pressurized at 700 psig. However, the pressure drop should be set to avoid the Thomson-Joule effect, i.e. expansion through sample valve that can lead to sample phase change and/or cavitation. Cavitation will damage the sample valve. Users must make sure that the pressure drop inside the sample stream selection or the GC sample injection valve will always remain higher than the vapor pressure of the liquid sample under the operating temperature. Having too much pressure drop on the sample valve will result in a too high sample velocity through it. This may cause other valve problems, like wearing of polymer material by erosion through high speed fluid flow generated friction. Never operate sample valve in these conditions. In order to prevent such situation, it is recommended to add a flow restrictor on the vent side of the GC sampling valve.



Avoid Thompson-Joule effect, cavitation and sample phase change.

- R1: Limit total sample flow in case of a diaphragm valve failure
- R2: Specifically tuned to limit pressure drop

The flow restrictor is tuned to minimize the pressure drop on the valve while having enough sample flow. It doesn't make sense to have a GC valve with an internal sampling loop of 0.5 microliter, and a sample flow of 1 or 2 sccm (2000 microliters). Typically, a valve having 0.5 microliter internal sampling loop will have an internal sample flow of about 0.3 sccm at 20 psi differential pressure. Here we are talking of liquid sample. With this sample flow through the valve, the sample loop volume is refreshed 600 times per minute, which is fast enough. If the liquid sample is at 700 psig at the valve inlet, and the return point into the process is at 300 psig, the flow orifice is set to have a pressure drop of 380psi at 0.3 sccm. So the valve is exposed to a safe 20 psi pressure drop on it while operating at 700 psig inlet pressure. You should not exceed the maximum specified static operating pressure of the valve. A valve could be operated under a relatively high static pressure for example 1000 psig, but the pressure drop between two connected valve ports must be kept in the safe operating pressure area of the valve. This pressure value is valve dependent and also related to the sample fluid thermodynamic data.

Now, if the sample lines are long, a specific sample by-pass flow control valve having a higher Cv must be installed before the sample stream selection system. The sample by-pass flow will be set at a value high enough to get the target speed of response. Normally in process GC, this value is about 90% of a step change at the sample line inlet, measured at the sample line outlet after 60 sec.

As a GC components manufacturer, ASDevices is getting many orders for valves with various configurations. In several occasions, for OEM or system integrator marketing reasons, we don't know where and how the valves will be used. The key to success of a process or laboratory GC analyzer is the reliability of the GC valve installed in it.

To get reliable valve operation, the configuration should be done to be compatible with the sample. Furthermore, the operating conditions like pressure, flow and temperature may play an important role in the valve final tuning, setting and installation. User must be sure that valve materials are compatible with the sample.

OVERALL SYSTEM CONCEPT

We divide our system in three separate zones ;

- **Zone 1** is the process point sample connection and sample line that carries it up to Zone 2.
- **Zone 2** is the sample stream selection system.
- **Zone 3** contains the analytical instrument i.e. a process gas chromatograph.

In each of these zones, special care in selecting the materials and also special considerations must be taken to allow safe and stable operation.

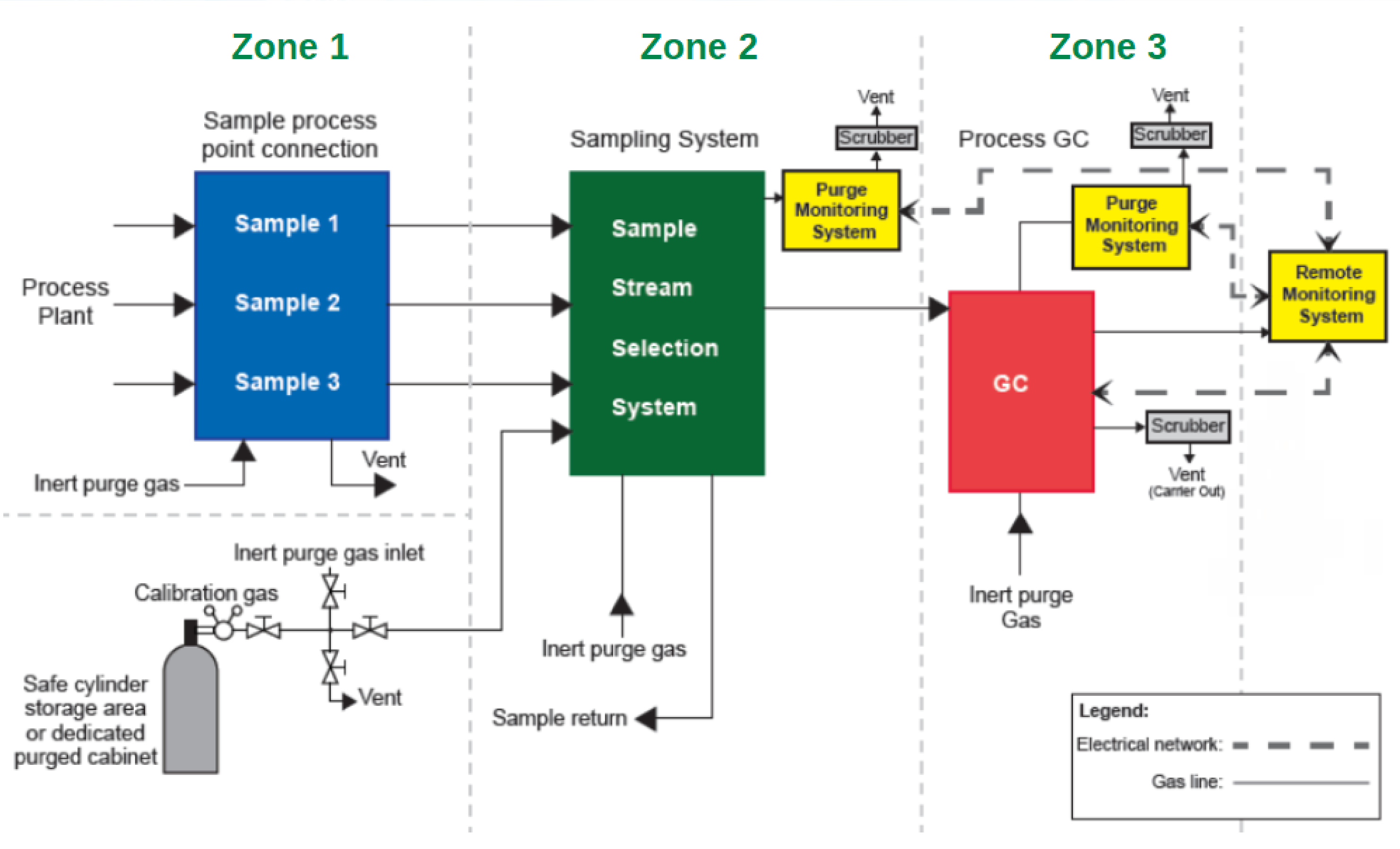


Figure 3—Schematic presenting the overall concept of the system with the three zones.

As shown in figure 3, we must first connect our sampling system (Zone 2) to sample connection point at various locations in the process (Zone 1). There are different compositions of silane or chlorosilane mixtures at various process sample connection points.

These samples must be analyzed for process control and reported as variables used to tune plant operating parameters or as quality control measurement. In this latter case, it is to ensure that the final product meets the required quality standard.

In our proposed system example, there will be three sample inlets coming from various locations in the process plant producing silane and chlorosilane. An inlet is also reserved for a calibration gas and another one for clean purge inert gas. See figure 4 for simplified schematic block.

A total of five inputs could be selected to flow into the analytical equipment. Since we are working with nasty and unfriendly sample gases, our philosophy is to allow this sample to flow into the system only when required. This way we are reducing the load or the working duty cycle on critical parts of the system, like sampling selection and injection valves. Most of the time, a sample is injected for about 30 seconds and the GC injection valve is returned to its sampling position, while waiting for the full chromatogram to be eluted and quantified. Depending on the GC configuration, it is not uncommon to have a GC run time of 5, 10 or even 15 minutes. So why keep dangerous gases flowing into the GC sample valve during this time? Furthermore with this concept, the response time of the system is not reduced, since there is sample flowing at all time in the sample lines.

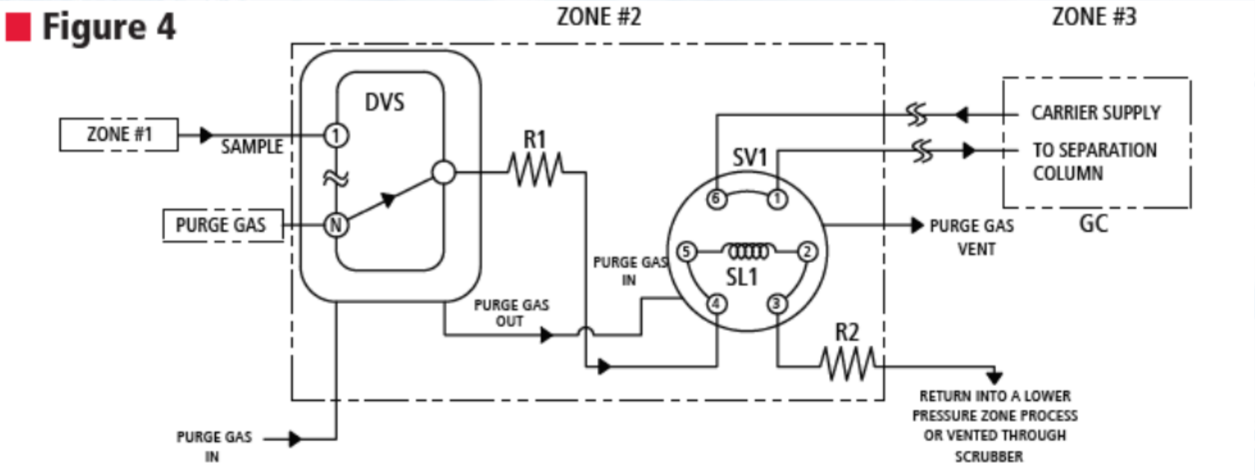


Figure 4—Block diagram of the basic concept.

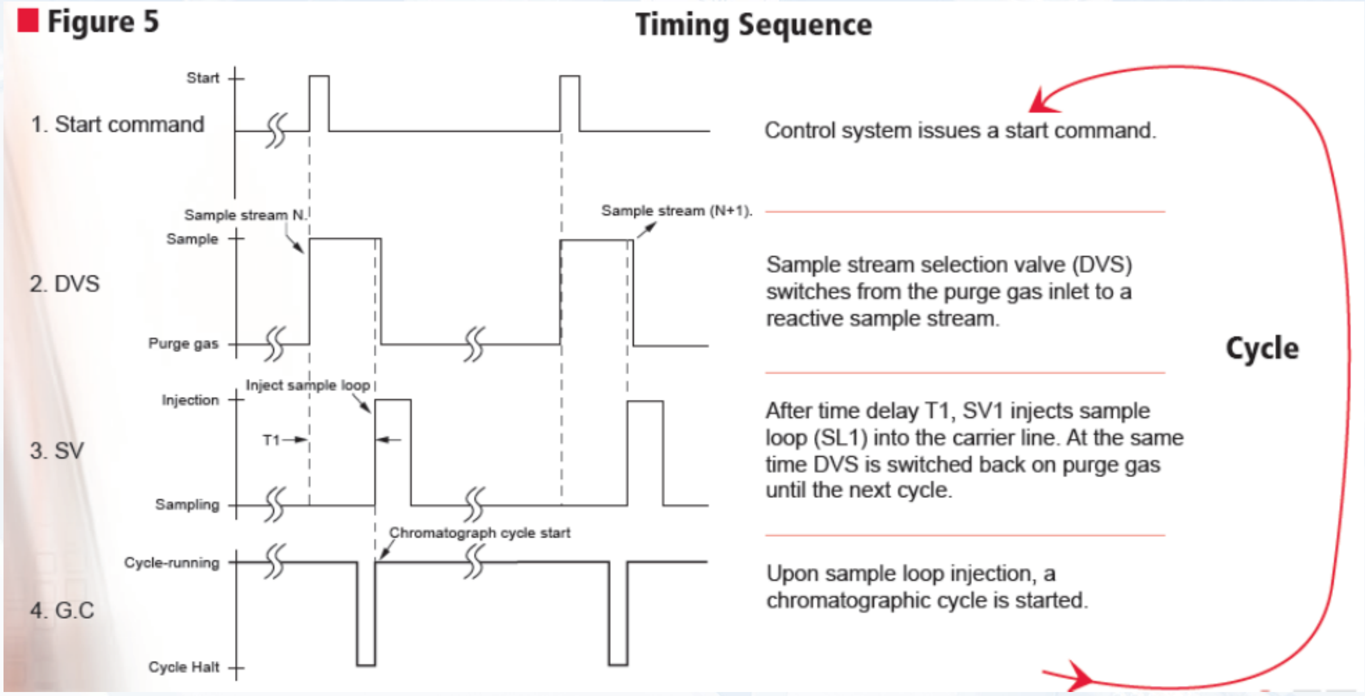


Figure 5—Timing sequence associated with Figure 4.

> *‘‘Instead of having a GC and associated sampling system exposed to dangerous compounds 100% of the time, the working time could be reduced to around 3%, depending on specific GC configuration. This results in lower risk since there are dangerous samples flowing into sample stream selection and GC valves for only 3% of the time. Furthermore, there is only a small discrete volume of sample allowed to flow into the GC at a time, which will have beneficial effects on lifetime of the components. Indeed, the fact that the critical hardware is less exposed to dangerous compound will extend their lifetime. Pure and clean inert gas is flowing through them the rest of the time.‘‘*

4.0 SYSTEM DESCRIPTION

There could be many variations in system configuration in order to achieve our goals. The one we propose here is based on our experience and could be used as a general guideline to design your own system. The idea behind the proposed concept is to minimize the flow of lethal and poisonous chemical compounds into the analytical equipment and, at the same time, to be able to sample many points accordingly to the process control needs. Later in this paper, we will describe other design variations that reduce even more system exposures, by placing another GC valve close to or in Zone 1, in a field station cabinet. Don’t forget safety is our priority when working with such samples.

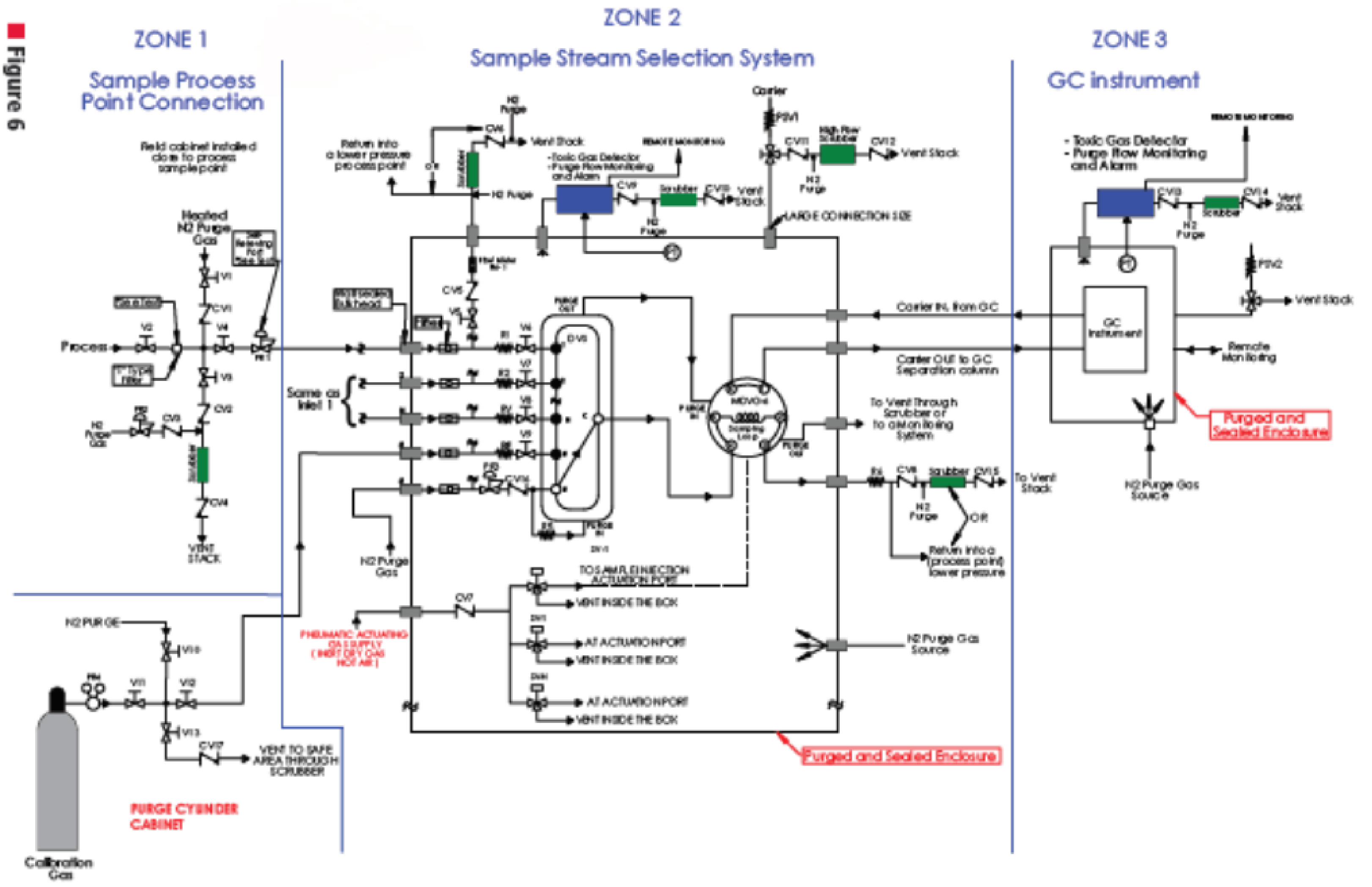
> *‘‘In some installations, a double wall sampling line is used. With such tubing, there is a purge flow in the coaxial area, i.e. between the outside diameter of the internal tube and the internal diameter of the external tube. This is normally governed by local regulations and/or by company safety code and policy. Monitoring the purge gas flowing into coaxial area for its pressure and quality gives an indication of leaking problems.’’*

ZONE 1 : PROCESS SAMPLE POINT CONNECTION

As we said earlier, we have divided our complete system into three separate zones. The first one is the process sample point connection. Depending on process pressure, temperature and phase, many variations could be done. A common procedure to do so is called the ‘‘cross T’’ purge system, see reference 3. This system allows the elimination of atmospheric contaminants before letting the reactive sample to flow through it. It also allows to remove the sample by purging with inert gas when maintenance is required. This avoids hazardous situation and it is a well proven system.

Figure 6 shows the process sample collecting system design configuration, in Zone 1 area. All valves, V1 to V4, are VCR or welded, PLSV. This is the same for check valves CV1 to CV4. No pipe thread fitting. Pressure regulator PR1 is a VCR or welded type, two stage, metal diaphragm based, self-relieving type. The self-relieving port must be connected to a vent line that is also connected to a scrubber. When the regulator is not self-relieving, the pressure at the outlet will eventually reach the process pressure value, if there is no flow through it. In some cases, this could result in a hazardous situation. Once again, this is application dependent.

The pressure regulator PR2 is selected to allow proper purge flow into the scrubber through CV3. All hardware in Zone 1 must be capable to work at a temperature up to 80°C or 100°C without problems. This is because we recommend to use a heated purge gas to speed up purging process and to eliminate moisture in a most efficient way. Cold purge gas will require


ZONE 1
Sample Process Point Connection
Field cabinet installed close to process sample point
Heated N2 Purge Gas
Process
CV1
CV2
CV3
CV4
V1
V2
V4
V5
Scrubber
VENT STACK
N2 Purge Gas
N2 PURGE
V10
V11
V12
V13
CV17
VENT TO SAFE AREA THROUGH SCRUBBER
PURGE CYLINDER CABINET
Calibration Gas
ZONE 2
Sample Stream Selection System
Return into a lower pressure process point
N2 Purge
Vent Stack
CV6
CV5
CV9
CV10
CV11
CV12
REMOTE MONITORING
- Toxic Gas Detector
- Purge Flow Monitoring and Alarm
Scrubber
High Flow Scrubber
Carrier
PSV1
LARGE CONNECTION SIZE
Same as Inlet 1
DVS
MDVO4
Sampling Loop
TO SAMPLE INJECTION ACTUATION PORT
VENT INSIDE THE BOX
AT ACTUATION PORT
CV7
N2 Purge Gas
PNEUMATIC ACTUATING GAS SUPPLY (INERT DRY GAS NOT AIR)
Carrier IN, from GC
Carrier OUT to GC Separation column
To Vent Through Scrubber or to gas monitoring system
CV8
Scrubber
CV15
To Vent Stack
N2 Purge
OR
Return into a (process point) lower pressure
N2 Purge Gas Source
Purged and Sealed Enclosure
ZONE 3
GC instrument
REMOTE MONITORING
- Toxic Gas Detector
- Purge Flow Monitoring and Alarm
CV13
CV14
Scrubber
Vent Stack
N2 Purge
PSV2
GC Instrument
Remote Monitoring
Purged and Sealed Enclosure
N2 Purge Gas Source

■ **Figure 6**

much longer purging time and much more volume of purge gas. It is a good practice to install a ''T'' type particle filter just after V2. ''T'' type filter can be easily cleaned when the system has been properly purged with an inert gas. This filter will trap particles coming from the process connection point. It will also avoid, in the present application, to get SiO2 powder into critical parts of the system. In case of process shut down or failure, which could be a possibility. Such filter body could be welded or installed with VCR fittings. Generally, there will be a welded pipe reducer from the process connection point to the sample line. Normally 1/4'' inch size sample line is used.

ZONE 2 : SAMPLE STREAM SELECTION SUB-SYSTEM CABINET

First, a solid steel enclosure, usually stainless steel, must be selected. This enclosure will hold all the related sample stream selection hardware, including pneumatic and electric valves, under a controlled inert atmosphere. This atmosphere is also monitored to detect trace of sample and report any hazardous gases. The internal pressure of the enclosure and the net exhaust purge flow is also monitored. Proper alarms will be generated in case of enclosure over/under pressure conditions or of a low purge flow situation. The over pressurisation of the sealed and purged enclosure is also protected by a pressure relief valve PSV1. PSV1 must be capable to allow high flow and its exhaust port must be connected to a safe vent header or through a scrubber for detoxification. The over pressurisation can be generated by a catastrophic valve failure or by a faulty purge gas pressure regulation.

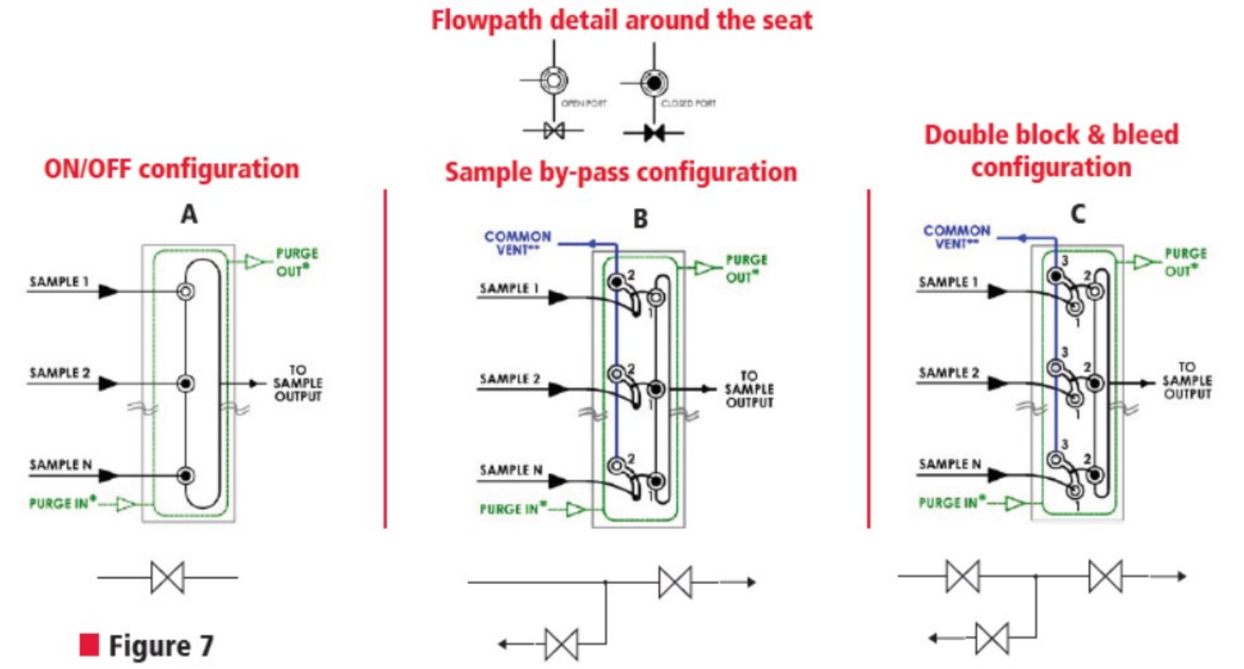
All gas lines and electrical cables are going into the enclosures through sealed feed-through connectors. There are many suppliers of electrical cable feed-through connectors in the market. The electrical feed-through connectors must be selected to avoid any back flow of gas into the electrical conduit or envelop surrounding various cables. For gas lines, appropriate wall sealed bulkheads are selected.

SAMPLE STREAM SELECTION VALVE

As a sample stream selection valve, we had selected a ASDevices µInProve sample stream selection valve based on the PLSV technology.This valve technology will last much longer than any other sample stream selection valve. With the PLSV technology, there is very little friction due to the reduced sealing force required. The sealing force is small because of the reduced surface sealing area. The wearing is very low and the technology is highly tolerant to particulates. This is not the case at all for a rotary valve where particles will be trapped between the rotor and stator surface. This will lead to cross ports contamination and eventually outboard leak. µInProve valves are very flexible in regard to the final application they will be used in. This is beyond the scope of this paper to attempt all possible variations. Basically the following figure 7 shows basic flow path configurations. In all configurations shown, there is a common feature: no dead volume effect or unsweep entrapment zone.

The PLSV technology offers other features that make such application easier. First, it offers a high level of sealing integrity. This is good to avoid cross-port contamination. With this valve, it is impossible to have cross-port leak due to the unique purge design. Secondly, the critical flow path is surrounded by a purged atmosphere inside the valve. All the wetted parts are surrounded by purged gas. The risk of inboard/outboard contamination is very low, almost impossible. These are critical points for those applications. Another benefit of the purge is the possibility to monitor the quality of the purge gas coming out of the purge vent. This allows early detection of fugitive emissions. This gives the possibility to have a very interesting benefit. Indeed, it provides a real time diagnostic system for the valve performance. For more information about application of the PLSV technology, see reference 5.

Each inlet is protected by a particle filter, in case of any SiO2 contamination coming from the external system. There is also a flow limiting orifice on each inlet. Those will limit the maximum total flow in case of a complete sampling gas chromatographic failure or sample stream selection valve major failure. This could happen if heavily contaminated samples are allowed to flow in these valves. All vents connected to the cabinet are going through an appropriate scrubber for detoxification. In many areas, this is imposed by environmental regulation.



■ **Figure 7**

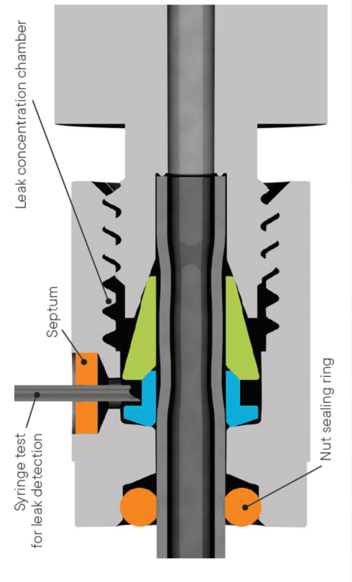
GC SAMPLE INJECTION VALVE

In this example, the GC sample injection valve is installed into the sampling purged cabinet. The sample coming out of the GC valve is going through a flow orifice in order to keep the pressure drop on the valve in a safe operating range. A high pressure drop will cause premature valve failure. The GC sample injection valve is a µInProve based on the PLSV technology. This is a valve with a complete embedded purge system. See reference 6 for more details [AN05 et AN08].

ASDevices µInProve injection valve with pneumatic actuator

FITTINGS

All unions and other compression fittings are coated LipLOK-based unions. The LipLOK design provides the sealing integrity of a VCR type fitting while having the flexibility of a compression fitting. The fine pitch thread and coated LipLOK characteristic of this fitting design require low torque to get very high level of sealing, as required by such application. The LipLOK uses two seal points, the first being the coated sealing ring, which is compression fitted to the tube end. The double ferrule design provides the second level of sealing, which resists the effects of vibration and protects against ejection of the tube from the fitting. Any leak developing inside the fitting will be forced to accumulate in a leak chamber. The pressure will build up in the chamber until a value where it will go to the sniffing port, which is sealed with a septum. Inserting the needle of a sniffer or leak detector allows sensitive leak detection since the leak is concentrated in the chamber. Such unique feature is especially interesting when working with highly toxic or reactive samples. See reference 7 for more details about the LipLOK.



ASDevices LipLOK

OTHER DETAILS

If sample by-pass flow valves are necessary (like V5), they could be controlled electronically or manually. Manual control will be done with a rotary feed-through mechanism or through magnetic coupling allowing to adjust the flow without having to open the door of the purge cabinet. The visual flow monitoring device is also mounted inside the purge cabinet. Obviously an appropriate sealed window is provided. Sample flow is by-passed through a check valve.

Each sample by-pass outlet could be connected together or separately connected through a scrubber and safe vent stack. Most of the time, they are returned into the process at a lower operating pressure point. This is application dependent; the system or process engineers will define the proper configuration for their needs.

ZONE 3 : ANALYTICAL EQUIPMENT PROCESS OR LAB GC

Dedicated process GC are easier to integrate and more appropriate for such applications. If laboratory or open frame GC are used for such applications, they must be under an appropriate ventilated hood, with an exhaust monitoring system. Some process GC come built-in into a purged enclosure ready to use. Some other process GC have general purpose enclosure but with an internal purge heated cabinet or oven. In all cases, the GC or other analytical instruments must be in a controlled and monitored environment to evacuate and report any hazardous leak. Generally speaking Zone 3 must have the same type of protection as Zone 2.

One problem we have seen with process or lab GC is the relative quality in regard to leak integrity of the electronic pressure controller (EPC). Many of them use miniature manifold mounted proportional valves. Such a valve uses O-rings for sealing against inboard / outboard contamination between the valve and the manifold. ASDevices has adressed this problem by designing a unique purged EPC. With this design, it is impossible to have inboard or outboard leak. The atmosphere around the EPC is continuously purged with inert gas, the same as the carrier gas. This greatly improve the analytical system performance and reduces hazards caused by leak. The same purged EPC can be used on the sample control line.

The regulation performance of such EPC are good and they are frequently used with FID and TCD detectors under air or N_2 as carrier gas. When using helium or hydrogen, there is a risk of atmospheric contamination. A few ppm of air diffusion will not significantly affect the performance of a FID or a TCD working in the percentage level. However, a few ppm of air diffusion into the carrier gas will be enough to cause important problems in our present application. Each time a sample is injected, it gets in contact with the contaminated carrier gas. More precisely, the outlet port of the injection GC valve, where the separation column is connected, will become corroded and/or SiO2 will be accumulated in this area (See figure 2A). Here, the purged EPC is consequently extremely important.

In some cases, users add miniature needle or metering valves to tune carrier flow. Don’t do this! Most of these fine tuning valves come with some type of stem seal or packing. This is an open door for inboard contamination. You don’t want to go to sea onboard a submarine with screen doors! There are some bellow metering valves, but their dead volume precludes their use for such applications. Use capillary orifices instead.

A WORD ABOUT SCRUBBER

All gases released into atmosphere must be detoxified. All reactive, toxic, lethal and flammable samples must be properly disposed. This can be done simply by returning the sample into a lower pressure point into the process. A pump could also be used if return pressure is too high. Different scrubber devices could be made. Neutralization could be accomplished with the help of an aqueous alkaline (basic) solution like soda ash, caustic, lime. Aqueous scrubber must have the proper size and must be maintained in working condition. Common flare stacks could also be used. In such case, all purge and sample vents are connected to a main header, where these gases are burnt or reacted with catalyst, in order to render them inactive. Chemical process engineers will be very helpful in configuring such devices and should be consulted.

5.0 STARTING UP THE SYSTEM

Proper start up procedures must be established, tested and followed. We will begin by purging all sample collecting points into Zone 1.

ZONE 1 START UP

Here we will explain the cross ‘‘T’’ purging procedure for one channel stream. The procedure is the same for all other cross ‘‘T’’ collecting points. PR2 is closed to shutdown scrubber purge flow. Refer to Figure 6.

1. Close V1 to V4;
2. Quickly crack open V1 to raise the cross ‘‘T’’ system internal pressure, and then close back V1;
3. Wait about 1 minute;
4. Slowly open V3 to depressurize the system until check valve CV2 and CV4 close. Then close back V3. This can be seen by checking for flow at the outlet of CV4. When internal pressure goes down below 20 psig (cracking pressure of CV2 and CV4), the check valves will close;
5. Repeat Step 2 to 4 inclusively about 15 times;
6. Open V1 and then V3 to have continuous flow of purge gas through CV2, scrubber and CV4. Let the purge gas flow in order to evacuate any trapped particles;
7. Open PR2, close V1 and then V3.

At this point-in-time, the internal cross ‘‘T’’ volume has no more atmospheric contaminant and the scrubber is kept under purge gas flow. Adjust the purge flow through the scrubber and CV4 high enough to avoid back diffusion of atmospheric air. This is what we call static purging procedures. The volume to be purged is purged by dilution instead of continuous flow. Having a purge gas pressure at 200 psig or even higher will accelerate the contamination dilution process. The use of vacuum pump connected on vent during start-up purging step will improve this process.

Furthermore having a hot dry purge gas helps a lot to get rid of moisture adsorbed on tubing internal surface. CV1 to CV4 are check valves with cracking pressure of minimum 10 psig. CV2 and CV4 make sure that there is no air diffusing back in the system when depressurizing it through V3. When the pressure goes below 10 psig into the scrubber, they close. Some installations have two check valves in series instead of one. Vent line must be vented in a safe area. Purge flow through vent line must be maintained.

In fact, it is a good idea to add another check valve with a minimum cracking pressure of at least 10 psig, just at the end of this line and before the safe vent connection point. This way vent line will still be pressurized with an inert purge gas and air is kept out, this is the role of CV4. This will isolate the scrubber from air in case of the absence of inert gas purge flow. The scrubber will detoxify the reactive sample before being vented. The choice of the scrubber material is application dependent. All scrubbers shown here must be properly selected in order to reduce the reactive compound sample below the safe value threshold.

ZONE 2 START UP

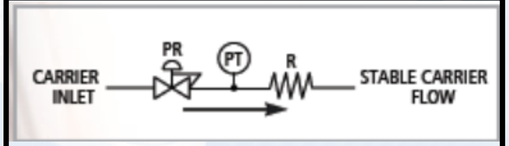
After having started-up Zone 1 sub-system like explained here above, we are ready to purge PR1 and the associated sample line. This is done by opening V1 and V4 in order to set a purge flow through PR1, the sample lines, V5 and CV5. The purge flow is supplied through V1 and CV1, while having V2 and V3 closed. This discharge pressure in swing between 10 psig (cracking pressure of all check valve in this configuration) and let’s say 100 psig. This step is repeated at least 15 times, over 20 to 30 minutes. This must be done while using hot purge gas. The number of steps and time required are system-dependent, i.e. sample line lengths and diameters. In some cases, it may be advisable to monitor the moisture level, with the appropriate moisture meter on the by-pass line outlet. When moisture level is below 1 ppm, there is no more air. Moisture level is a better indication, since moisture is absorbed more strongly than O2 on sampling system internal surfaces. When this step is done, PR1 pressure may be adjusted to an acceptable purge gas flow valve in order to maintain all the system under purge. Sample by-pass is set in order to monitor a net flow through the sample by-pass flow meter. This is adjusted with the help of V5. Keep the system under purge flow.

At this time V6 could be opened and the uInProve sample stream selection valve is switched to sample stream #1. This will allow the purge gas to flow through uInProve valve inlet #1, come out from the ‘’C’’ port, through uInProve PLSV Injector valve and sample loop, and be vented through R6, CV8 and scrubber. This process will evacuate air contamination. This process could be stopped when moisture level measure after CV8 is well below 1 ppm. At this point, set the purge flow through the sample injection valve at about 100ccm and keep it like this.

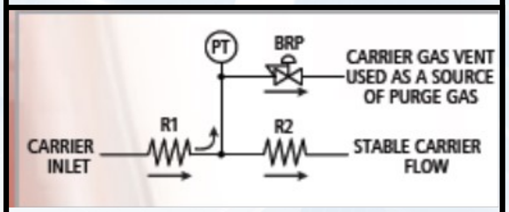
Every step of the above procedure must be repeated for each sample point stream. It is recommended to have an adjustable pressure regulator installed before V1 and a pressure gauge or sensor. This allows easy adjustment of pressure of purge gas as necessary during various purging steps.

When done, the same must be done with the calibration gas stream and the purge system. PR3 pressure regulator is set at the proper pressure to get the purge flow through the uInProve valves internal purge system. The valve purge vent could be monitored for diagnostic and / or vented through a scrubbing device.

ZONE 3 START UP



‘’Direct pressure / flow regulating scheme requires a leak-free and minimal dead volume proportional valve or pressure regulator. Orifice is tuned to work in sonic mode’’



‘’Indirect or by-pass pressure / flow regulation scheme could use the standard manifold mount miniature regulating valve as found in many GC electronic pressure controller’’

The GC must always be installed and started up as per manufacturer instructions. When carrier gas is helium, argon or nitrogen, we recommend the use of an heated double stage purifier based on a zirconium alloy such as the ASDevices ASDPure or iPaps. This purifier and associated hardware, like valves, fittings and tubings, must be selected to avoid air diffusion. Proper design will allow easy carrier gas replacement without polluting the purifier. Such purifier will meltdown if a slug of air is allowed to flow into it. This can result into a hazardous situation. Indeed, molten getter alloy could be propelled by the action of pressurized carrier gas. Such situation happened at some air separation plant analyser shelters. Some air separation plant safety policy imposes that such purifiers are installed in a protection cabinet to avoid such situation.

Having a pure carrier gas connected to the GC, we must make sure to keep it pure like this inside the GC too.

User must make sure that the internal electronic pressure controller is totally leak free. This is why ASDevices purged EPC is used in our case. In case of doubt, by-pass it and control the carrier pressure by other means. A safe way to do so is to use a miniature pressure regulator with a stainless steel diaphragm connected to a capillary flow orifice. The pressure regulator is stabilizing the pressure at the capillary flow orifice inlet. It is easy to tune the flow of a capillary orifice with the help of a bubble flowmeter. Such orifice will be a length of 1/16’’ OD tubing having an ID of .005’’ or .010’’. A section of this tubing is placed is a vise or similar equipment in order to ‘’pinch’’ it, i.e. to slowly reduce the internal cross section of the tubing while monitoring the flow at the desired inlet pressure set by the pressure regulator. Generally speaking, it is a good practice to set the absolute orifice inlet pressure 3 to 6 times its outlet value. Operating in these conditions would result in a velocity through the orifice that would reach the speed of sound or the sonic regime. At this value, if the inlet pressure is changed, the speed of fluid will stay the same through the orifice, only the gas density would change. It results in a very stable flow that is not affected by downstream back pressure changes.

Furthermore, such installation is leak free. No O-ring involved in the critical flow path. It is also easy to monitor the flow by monitoring the orifice inlet pressure. In sonic mode, the flow is linear with the inlet pressure. A simple look up table could be made in the system software or a simple relationship between pressure and the resulting flow could also be defined. This is the way we did it for many systems and it results in an exceptionally stable carrier flow for packed columns and stable velocity for capillary ones.

An even better way to regulate the carrier in a GC is called the indirect or by-pass method. This method fits well with AS-Devices' purged valves. The by-pass method requires to vent some carrier flow. This carrier flow is then used as a source of purge gas for various diaphragm valve in a GC. Furthermore, this method has the benefit to use very basic proportional valve or back pressure regulator, as long as there is no air diffusion into them. Another benefit of this method is the extreme stability and repeatability.

We did use this method in several systems with great results. With this concept, there is no flow control element in the carrier network, so no contamination. Both of these methods, i.e. direct or indirect, provide excellent carrier flow regulation in the GC. This is not the scope of this paper, but normally we use a flow sensor on carrier vent. Between the GC cycles, the system reads the flow and readjusts it (if needed) before to start another cycle. This is done by sending another pressure value set point to the direct or indirect pressure regulator. The system has a lookup table to convert flow in pressure. This is fast, precise, reliable and leak free. We did many systems using the indirect method to control the sample flow into a GC sample valve. It is also possible to do so in this application. Doing so, it becomes easy to monitor by-pass sample flow value. See annex B for a schematic of a GC configuration using this technique.

Having done all the necessary to establish a stable contaminant-free carrier gas and having the GC being started up, it is mandatory to start the purge system of the process GC or to close the hood of the purged cabinet of the laboratory GC. Again here same philosophy applies as per Zone 2, proper monitoring and detoxification system must be installed.

OPERATION MAINTENANCE AND TRAINING

The system is ready to be used by trained personnel. Training is part of the final system design. Operation and maintenance procedures must be available in writing and be audited and revised as the field situation evolves. Having a good sampling system is not enough, operation procedures must be followed. This is the key to success. Maintenance and hazard management are of prime importance.

Maintenance schedule or verification periods have to be routinely planned. Scrubbers and monitoring system have to be verified during these planned maintenance periods. System operation will vary depending on your GC manufacturer. It may come equipped with software having capability to control external devices and to build custom user interfaces. With such GC, it is easy to interface control and monitor the entire sampling system and also at the same time doing the chromatography task, i.e. column switching peak integration and reports. There are only few manufacturers that have these built-in capabilities.

With all the tools available today, cost is no longer an issue.

DESIGN CONCEPT VARIATION

The concept we propose here reduces substantially the system exposure to hazardous compound. Typically the exposure is about to 3 % to 5%. This is achieved with a proper system timing. This concept extends the overall system lifetime and reduces dramatically the risks of hazardous situation by reporting them before they happen. It is still possible to reduce the sampling system exposure to these chemical compounds. Here we are talking about the sampling tubing lines that carry the sample back and forth to the sample stream selection system in Zone 2. Indeed in our proposed concept there is a flow of hazardous compounds at all time in these various tubing lines. The length of these tubings could be quite long depending on location of sample process connection points and the sample stream selection system, i.e. the distance between Zone 1 and Zone 2. These tubings also need protection to avoid to get pinched or broken. The following design concept variation resolves this issue. Figure 8 is showing a concept where a GC valve SV1 and its associated sample loop are mounted in a field station very close to the Zone 1.

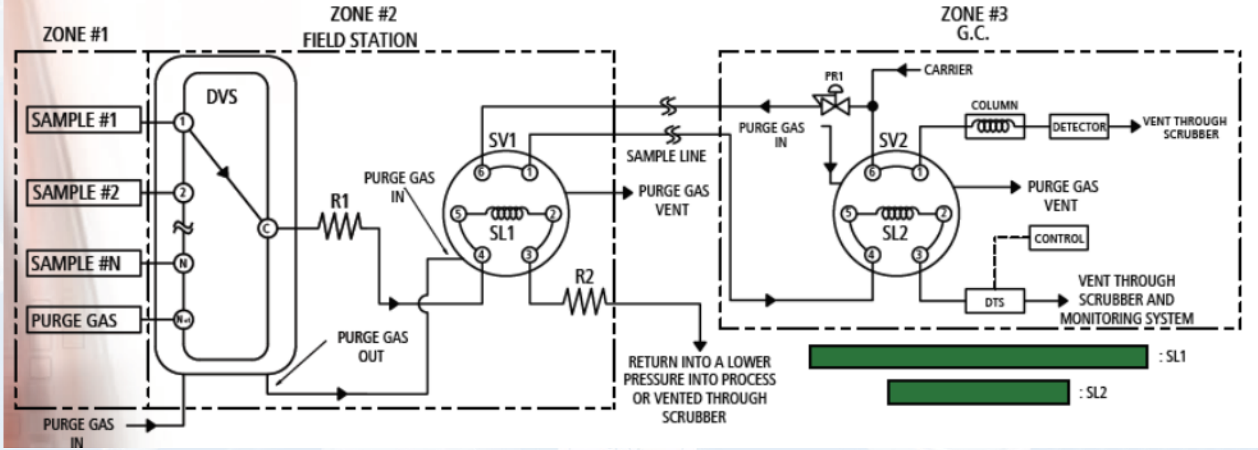
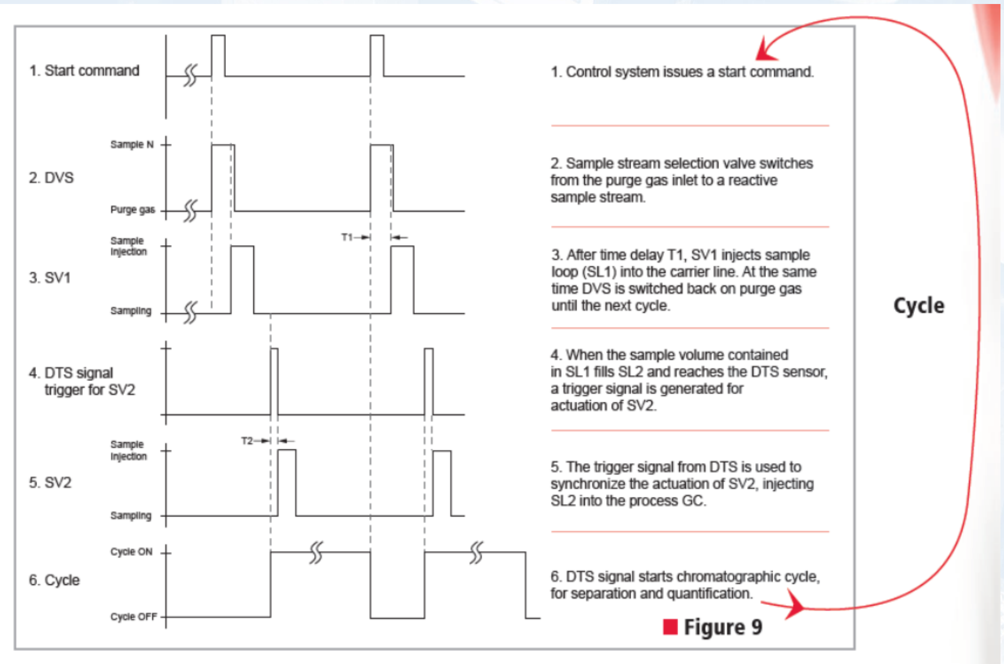


Figure 8—Concept where a GC valve and its sample loop are mounted in a field station close to Zone 1.



1. Control system issues a start command.

2. Sample stream selection valve switches from the purge gas inlet to a reactive sample stream.

3. After time delay T1, SV1 injects sample loop (SL1) into the carrier line. At the same time DVS is switched back on purge gas until the next cycle.

4. When the sample volume contained in SL1 fills SL2 and reaches the DTS sensor, a trigger signal is generated for actuation of SV2.

5. The trigger signal from DTS is used to synchronize the actuation of SV2, injecting SL2 into the process GC.

6. DTS signal starts chromatographic cycle, for separation and quantification.

Figure 9

Another GC sample injection valve SV2 is located inside the GC in Zone 3. Sample loop 1, i.e. SL1 size is larger than the sample loop 2, i.e. SL2 located on the GC valve SV2 inside the GC, SL2 could be internal or external mounted into SV2 as long this one is much smaller than SL1. There is a thermal conductivity sensor, i.e. DTS, on the sample vent of the GC valve SV2.

An independent control system or the software inside the GC sends the sample injection command to SV1. The carrier gas carries the sample slug or aliquot of SL1 through SL2. SL1 has a larger size than SL2 making sure that SL2 is really filled with the sample at the middle of the sample slug length. This reduces the effect of sample fragmentation that may happen on the boundaries of the slug. With this system, we have seen no sample separation, band or time broadening. However, user must take into account the thermodynamic behavior of the sample. Depending on the boiling point of a particular sample, it may be necessary to use probe for sample extraction, heated sample lines, vaporizer, etc. This is true for mixtures having a large range of boiling point or for highly polar molecule impurities. The sensor DTS will detect a drastic change in thermal conductivity. This is used as a trigging signal for injecting SL2 into the GC separation column.

PR1 sets the carrier flow velocity through SL1. The timing is easily adjusted by monitoring the signal from the sensor DTS. The speed of response of the sampling lines could be adjusted by setting PR1. The field station box follows the same rules in regard to the use of a sealed and purged enclosure and the associated hardware, i.e. scrubbers and monitoring system. Some users also add contact switches on various enclosure doors to detect enclosure opening and act accordingly.

OTHER VARIATIONS

A sampling technique having a concept close to what we propose here has been done by Jimmy G. Converse, in the eighties. They named it ''Remote Discrete Sampling''. See references 8, 9 and 10 for description of such system. The difference between this concept and ours is the fact that we switch the system on purge gas between the cycle, reducing even more the sampling system exposure. The references 8, 9 and 10 are interesting and well done. They point out, like we do, the necessity for safety and for reducing system exposure, this is obviously not a new problem.

> ''**In Short:** Never allow continuous flow of hazardous sample through a GC valve mounted directly into an instrument. This will eventually end into a potentially dangerous situation.''

REAL-TIME VALVE DIAGNOSTIC

The remote monitoring system PLC controller will report any problem with the status of the sampling system and the environment where the process / lab GC is installed: purge flow, internal sealed enclosure pressure, hazardous compound leaking into various seal boxes, etc. In any case, an extra step could be done into this direction, resulting in monitoring valve health in real time.

Indeed, the internal valve purge flowpath surrounds all wetted parts of the PLSV technology. So any sample leak or outboard of the valve will be captured by the purge flow and carried away at the valve purge outlet port. It is not uncommon for the users to monitor this purge flow coming out of the valve purge vent ports.

Some users add an extra valve to re-inject, with a pre-defined programmed time, and guide this purge gas into a chromatographic channel to verify the quality of this gas. If something is wrong, an alarm is issued, warning the operator about potential valve failure.

In case of early problem detection, maintenance could be planned instead of unexpected system shutdown.

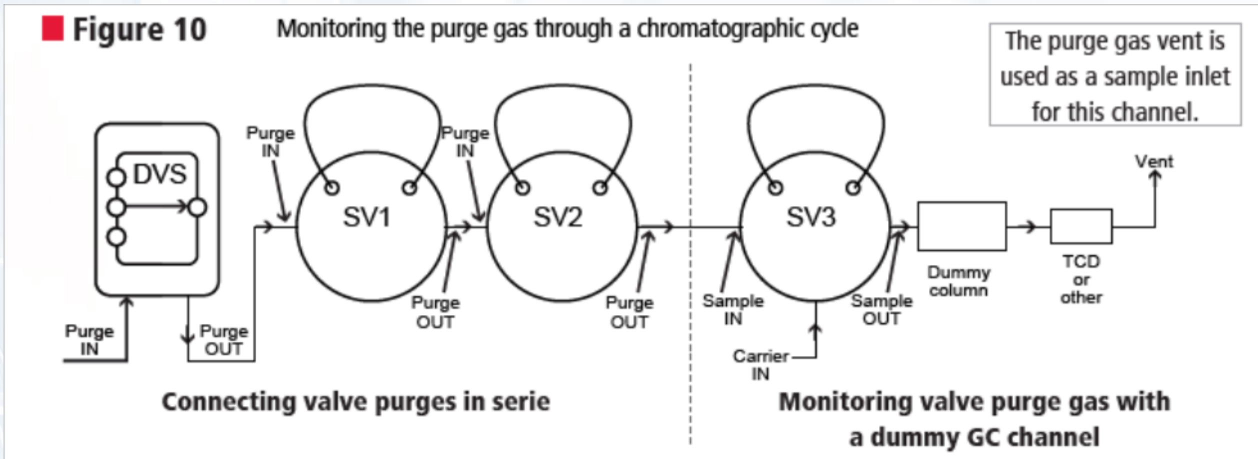


Figure 10—Monitoring the purge gas through a chromatographic cycle

EXTRA INTELLIGENCE AND PROTECTION

With today's availabilities and low cost of independent discrete control systems like PLC, i.e. programmable logic controllers, it is easy to automate all the sampling process. Pressure flow and temperature sensors are relatively inexpensive and directly interfaceable with PLC. User's interface, network capabilities, etc. are, by today standard, inexpensive. With such tools, it's relatively easy to configure proper system interlock, monitoring and alarming. Alarming reports can be sent to any computer, laptop or smartphone. The use of backup power supply is mandatory in order to maintain the functionality of the monitoring and control system, in case of power failure.

Another design variation will be to automate all the valves in Zone 1 and in case of any toxic gas detection in various purge cabinets i.e. the sample stream selection or GC cabinet, proper valve operation will stop sample flow and allow the system to switch on an inert purge gas. Cylinder valves could be also easily automated. There are pneumatic operated cylinder valves that need air pressure to keep them open. Such valves are used with flammable, corrosive and/or toxic gas cylinders. See reference 11 for more information about cylinder valves. It is also a good practice to install limiting flow orifice at the outlet of these cylinder valves.

TYPICAL SYSTEM RESULTS

Figure 11 shows a customer reported chromatogram, with a sampling system deeply inspired by our proposed concept. This system is in used since nine years and the first valve maintenance was done one year ago. No incident reported. Accuracy and repeatability still inside the system specifications.

This is due to the fact that the system is exposed about 4% of the time to aggressive media. Proper sealed and purged enclosures are used and monitored. This system reports ppm impurities in pure silane. GC configuration has a few packed columns and ionisation detector.

Figure 12 shows another chromatogram involving chlorosilane composition. Again here, the concept following the sampling system described in this paper has been done. This system is in use since several years, without incident or need for valve replacement. This GC system has a few columns, one TCD and one FID.

The new technologies developed since then by ASDevices, such as the purged EPC, the PLSV valve technology, LipLOK fittings, etc., which are used in the iMov platform, can only be beneficial for the lifetime of such analytical system.

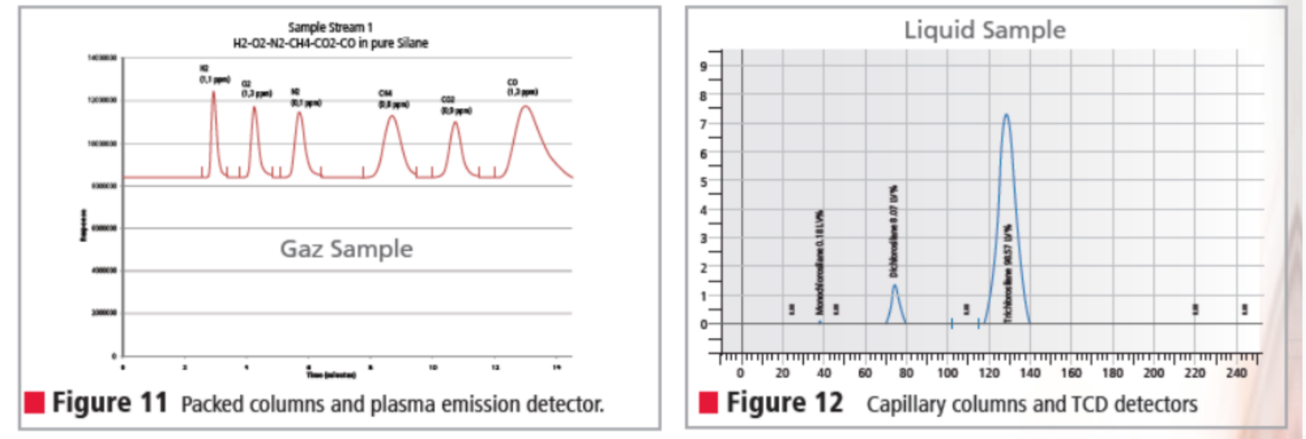


Figure 11 Packed columns and plasma emission detector.

Figure 12 Capillary columns and TCD detectors

Figure 11-12—

Another application results using the same design sampling philosophy is shown in Figure 13. This chromatogram show benzene and toluene level in gasoline sample.

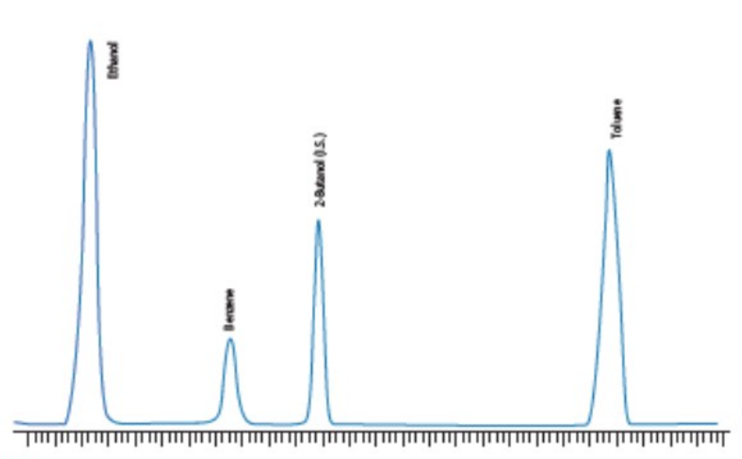


Figure 13— Gasoline Sample

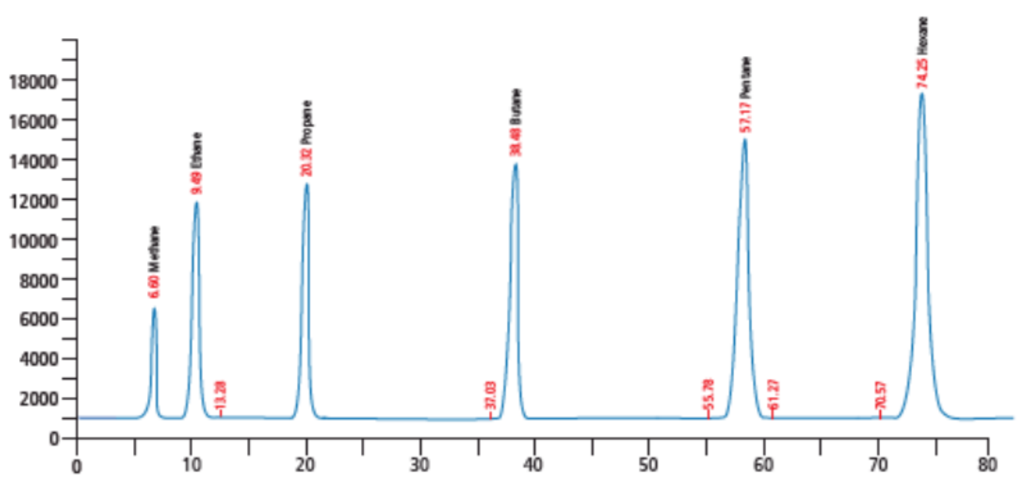
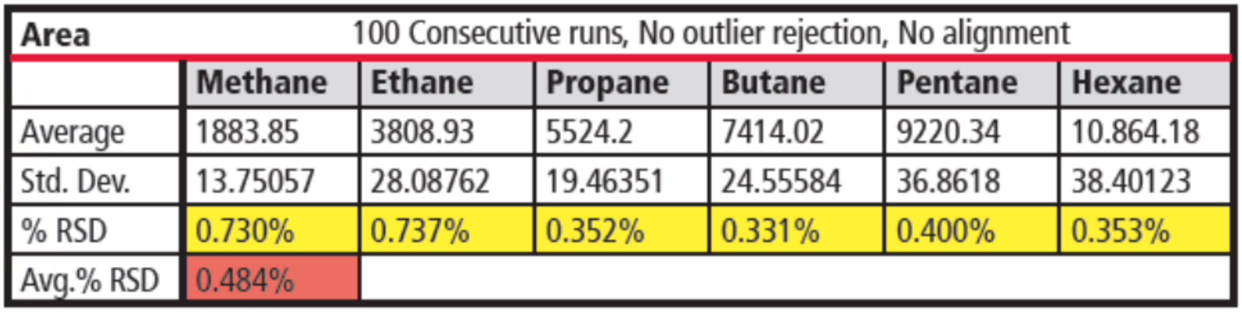


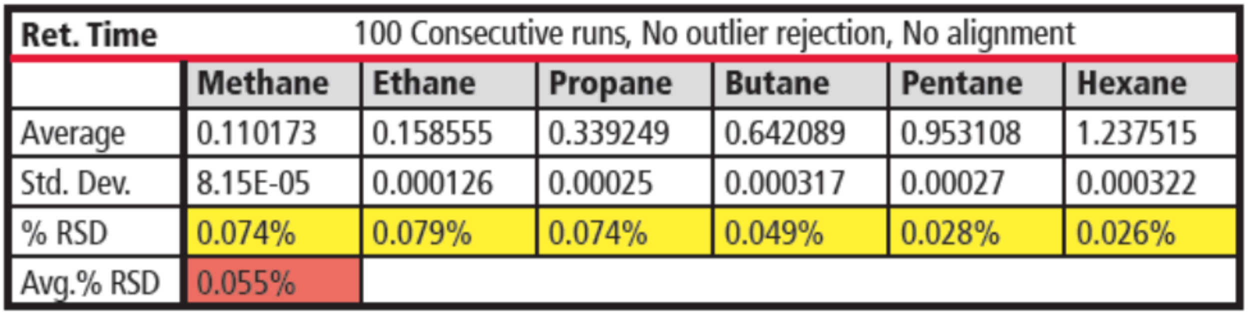
Figure 14— 100 consecutive samples overlaid, C1 to C6

Figure 14 shows system overall repeatability measurements done from C1 to C6. The GC valve was a 6-ports µInProve using the PLSV technology. The test was done with 100 consecutive measurements.



Area	100 Consecutive runs, No outlier rejection, No alignment					
	Methane	**Ethane**	**Propane**	**Butane**	**Pentane**	**Hexane**
Average	1883.85	3808.93	5524.2	7414.02	9220.34	10.864.18
Std. Dev.	13.75057	28.08762	19.46351	24.55584	36.8618	38.40123
% RSD	0.730%	0.737%	0.352%	0.331%	0.400%	0.353%
Avg.% RSD	0.484%					

Table 1— Measured area for 100 consecutive runs, no outlier rejection, no alignment.



Ret. Time	100 Consecutive runs, No outlier rejection, No alignment					
	Methane	**Ethane**	**Propane**	**Butane**	**Pentane**	**Hexane**
Average	0.110173	0.158555	0.339249	0.642089	0.953108	1.237515
Std. Dev.	8.15E-05	0.000126	0.00025	0.000317	0.00027	0.000322
% RSD	0.074%	0.079%	0.074%	0.049%	0.028%	0.026%
Avg.% RSD	0.055%					

Table 2— Retention time for 100 consecutive runs, no outlier rejection, no alignment.

6.0 CONCLUSION

The cost associated to build a good and intelligent sampling system pays back by itself. Indeed, many years of trouble free service are possible, even with such challenging samples. The concepts that have been described in this paper are those ASDevices recommends and asks to follow when working with lethal or very reactive samples. Any other way is not approved or supported by us. We consider these as a minimum requirements.

The main idea behind various concepts proposed is to keep away the nasty samples, as much as possible, from all parts of the system, including the personnel. This reduces dramatically the risk and improves the life time and system reliability. Maintenance is also reduced significantly.

There are many variations or modifications that could be done to reflect your needs. We believe that such system designs must not only involve the analytical chemists, but also instrumentation and plant process engineers. With all this and some creativity, innovation, ingenuity and some common sense as basic ingredients, it will result in a safe and reliable design. Know the regulatory requirements. Don't hesitate to contact Analytical Sensing Devices for assistance in your design.

FURTHER READINGS

We invite you to pursue your reading by consulting reference 14. It provides a check list and suggested safe practices while handling toxic and hazardous gases.

ACKNOWLEDGMENTS

The ASDevices team would like to thank customers who did accept to share with us their comments and results. Unfortunately and for obvious reasons, they don’t want to have their name disclosed and we respect this.

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The technology described herein may be subject to patent protection or other forms of intellectual property right.

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ANNEX A - SOURCE OF USEFUL INFORMATION

1) **US Silane Codes and Standards**

NFPA 318 & 55 (National Fire Protection Agency)
IFC Chapters 18, 27,30 & 41 (International Fire Code)
FM Global 7-7 (Factory Mutual)
ANSI/CGA-G-13 (American National Standards Institute/Compressed Gas Association)
SEMI S-18

2) **Silane Standards**

Two standards for Silane are being adopted worldwide

- SEMI S18-1102 Environmental, Health and Safety Guideline for Silane Family Gases Handling
- CGA G-13
  Adopted as an ANSI standard.
  Adopted by the US Fire Codes as the silane safety standard

It is being adopted as a global standard by European, Industrial Gas Association (EIGA), Asia Industrial Gas Association (AIGA), and Japan Industrial and Medical Gas Association (JIMGA)

3) **Scope and Purpose of CGA G-13**

Addresses the hazards in handling silane
Covers storage, use and cylinder filling facilities
Prescribes control for installation of silane systems
Recommends methods for storage & transfer of silane
Provides guidance for sitting, design of equipment, safety systems & installation of silane storage and gas delivery/filling systems
Provides guidance on operational steps
Does NOT cover user facilities and gas distribution after the gas cabinets or VMB's

4) **Silane related regulatory**

FEDERAL:

- Department of Transportation (DOT), Regulations for Flammable Materials
- Environmental Protection Agency (EPA), The Clean Air of Act of 1990
- Occupational Safety and Health Act (OSHA), Process Safety Management

STATE and LOCAL:

- Uniform Building Code (UBC) 1991, Chapter 9, Hazardous Production Materials
- Uniform Fire Code (UFC) 1991, Articles 10, 51, 80

5) **Industry standards and guideline for silane**

⍰ **Compressed Gas Association**:

- Standard V-1, Compressed Gas Cylinder Valve Outlet and Inlet

⍰ **Semiconductor Equipment and Materials International (SEMI)**:

- SEMI F1-90, Specification for Leak Integrity of Toxic Gas Piping
- SEMI F2-94, Specifications for 316L Stainless Steel Tubing for General Purpose
- SEMI F4-90, Guide for Remotely Actuated Cylinder Valves
- SEMI F5-90, Guide for Gaseous Effluent Handling
- SEMI F6-92, Guide for Secondary Containment of Hazardous Gas Piping Systems
- SEMI F13-93, Guide for Gas Source Control Equipment
- SEMI S5-93, Safety Guideline for Flow Limiting Devices
- SEMI S6-93, Safety Guideline for Ventilation

6) **Occupational Safety and Health Administration (OSHA) USA**

Note : The above list are useful source of information issue at the referred publication date.
Please see your local agency for up-to-date regulation in handling hazardous matters.


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