Technical White Paper

Sampling Liquid Petroleum Gas

(and other high vapor pressure gas/liquids)

I. Purpose

The primary purpose of this technical note is to provide insight into the history and issues related to the sampling of Liquid Petroleum Gas (LPG) and other high vapor pressure liquids and to provide recommendations for accurate, safe and environmentally compliant sampling methods.

II. Why Sample Cylinders?

Sampling LPG and other 2-phase liquids that have high vapor pressures require sampling techniques using a metal cylinder (sample cylinder) for collection and transport. For clarity of discussion, we assume that a sample cylinder is an assembly that includes a pressure rated cylinder and inlet and outlet isolation valves as a minimum. They can be used in open-ended or closed loop (preferred method) sample extraction methods.

Sample cylinder assemblies provide several advantages for this type of sample: 1) they allow for samples to be collected at a pressure, at or near the pressure of the process being sampled. This assures that the sample constituents that can change state with pressure reduction are not lost during sample collection. An example of this type of sample would be LPG or other two-phase fluids that could flash off sample constituents if the pressure was reduced during sampling. 2) Since the sample is a pressurized liquid that has a high vapor pressure, the sample cylinder assembly provides for a safe method of collection and transport as long as the maximum fill density meets regulatory requirements. See definition of maximum fill density.

III. Definitions

In order to provide a common base of understanding, the following definitions are set forth and used in this document. They are not intended to set any standards, but just to make discussion of sampling processes and techniques clearer.

A. Sample Cylinder Assembly - A metallic cylinder (typically stainless steel) designed to varying pressure levels, inlet and outlet valves to isolate sample within the cylinder and some connection method for easy removal of the sample cylinder assembly from the sampler. Additional options might include blow out (rupture) discs or pressure relief valves, handles, identification rings, bypass lines, etc. Outage tubes are sometimes used in sample cylinders. See definitions for outage tube and maximum fill density below.

B. Maximum fill density - The volume of a sample cylinder assembly usually expressed as a percent of total capacity that is regulated by various governmental authorities for safe transport of the cylinder. International regulations sometimes vary, but most limit the maximum fill density of transport containers for safety to 80% of liquid fill at 15°C. For further information of these regulations you can reference U.S. CFR49, Canadian Transportation of Dangerous Goods Regulations, individual country transport regulations and IATA regulations regarding the limit of percent fill of containers used in shipping LPG. Since sample cylinder assemblies can be transported for analysis and for safety in general, most end users sampling LPG and other high vapor pressure liquids typically follow maximum fill density regulations.

Compliance with fill density requirements, when done after the sample cylinder assembly is removed from the sampler, presents problems for representative sampling, operator safety and environmental compliance. A variety of methods to solve this have been developed over the years, some better than others. These various methods are the real difference to evaluate when selecting a sampling method for LPG and other high vapor pressure fluids. The various methods will be discussed below with specific comment as to their ability to provide a representative sample, maximize operator safety and reduce or eliminate discharge of VOCs from the sampling process thereby meeting applicable environmental regulations and minimizing waste.

C. Outage tube - a “cut to length” tube placed inside of the sample cylinder used as a way to remove excess sample from the cylinder via manual evacuation after the sample cylinder assembly is removed from the sampler. **Note: Some misconceptions in the use of outage tubes presume that by merely having an outage tube in the cylinder you can achieve the 80% maximum fill density. This is not true. The product pressure fills the sample cylinder to the point where the initial gases in the cylinder are compressed to the same pressure as the liquid. This point is well above the outage tube end. Getting to the maximum fill density by use of an outage tube requires manual release of excess liquid as noted below.** ASTM 1265 method suggests that the outlet valve on the cylinder (end with the outage tube) be opened to allow escape of fluid down to the dip tube length to meet the maximum fill density. This is done by the operator slowly opening the outlet valve on the assembly and discerning when liquid stops coming out the assembly by listening for “sputtering” as the change if flow behavior goes from liquid/vapor mix to just vapor.

D. Vapor Pressure - the pressure exerted by a vapor; often understood to mean saturated vapor pressure (the vapor pressure of a vapor in contact with its liquid form). The higher the vapor pressure of the vapor within the liquid being sampled, the greater the propensity for the liquid to change state as its contained pressure is lowered.

IV. Sample Connection Types (in order of those typically used today)

Speed loop sampling connection with venting provision - Two connections to the product line at different pressures (usually without phase change) that provide for the flow of product through the loop and the sampler. There is either centralized or local collection/processing of off gases or vapors released during the sampling process eliminating product release to the local environment and personnel. This does not mean that after the sample cylinder is removed from the sampler that there is not release of vapors. This is dependent on the type of sampling process used (outage tube, expansion vessel or sight glass).

Single point sampling connection with venting provision - A single connection to the product line that the sampling device is connected to. No product return line exists. There is either centralized (vent or flare header) or local collection/processing (charcoal canister) of off gases or vapors released during the sampling process eliminating product release to the local environment and personnel. This does not mean that after the sample cylinder is removed from the sampler that there is not release of vapors. This is dependent on the type of sampling process used.

Single point sampling connection without venting provision - A single connection to the product line that the sampler is connected to. No product return line exists. There is no provision for centralized or local collection or processing of off gases or vapors released during the sampling process. Off gases or vapors that are released during the process go to the local environment and personnel.

A. Speed loop sampling connection with venting provision

This is the most common of sampling techniques for LPG and other high vapor pressure liquids when a speed loop is possible. Product loss during sampling is low since product is recovered during the sampler purging, and, in some methods when the maximum fill density is being achieved. Several methods have been used over the years and each will be explained.

1. Speed loop using a sample cylinder assembly with an outage tube

   This method has been used for many years and provides the advantage of product flow through the sampler and the sample cylinder assembly to improve sample representativity. Its typical operation (shown with the use of a Sentry Tandem Switching Valve) is shown in figure 1. Other versions use multiple valves to accomplish the same result and therefore add complexity and maintenance.

Legend:

1 = Sample inlet connection
2 = Sample outlet connection
3 = Vent connection

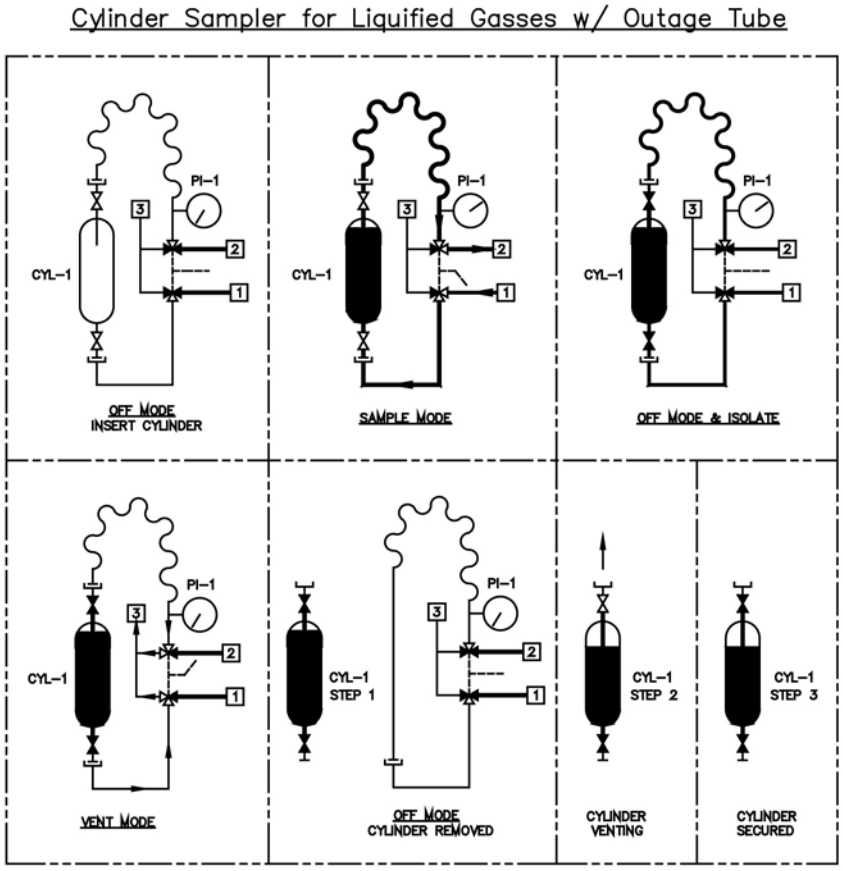

Figure 1

Off Mode (insert cylinder) - The tandem switching valve handle is turned to the off position blocking both side ports on both 3-way valves on the switching valve. There is no flow through the sampler. An empty sample cylinder assembly with outage tube is placed into the sampler in this mode in preparation for sampling.

Note: Sentry samplers also use a male and female Quick Disconnect (QDC) set on the sampler (also required on the sample cylinder) so that when the sample cylinder is not on the sampler, the fixed QDC and the hose QDC can be connected to close the sample loop on the unit. This prevents damage to the QDC ends, keeps them clean and minimizes the number of sample cylinders needed for the sampling process.

Sample mode - The tandem switching valve handle is turned to the sample position and sample flows through the sampler and the sample cylinder assembly. This is essentially a filling/ purging process. Notice that on LPG (and liquids) the sample inlet to the cylinder is on the bottom. This allows the liquids to push out any old sample or air to the sample return up to the point that the sample reaches the bottom of the outage tube. **Note: At this point, remaining air or other gases/liquids are trapped in the upper part of the sample cylinder assembly above the outage tube and are compressed by the sample pressure.** The pressure gage shows that system pressure is on the sampler. It does not directly indicate sample flow, but provides an indirect indication that sample has reached this point.

Off and Isolate mode - The tandem switching valve is turned to the off position to block sample flow. Then the isolation valves on the sample cylinder assembly are closed isolating the sample.

Vent mode - The tandem switching valve is turned to the vent position and product in the sampler lines above and below the sample cylinder assembly are vented to the vent line. This process is very efficient in eliminating these gases/liquids in the sampler lines for LPG since the vapor pressure of LPG is substantially above atmospheric pressure.

Off mode (remove cylinder) - The switching valve handle is turned to the off position blocking both side ports on both 3-way valves on the tandem-switching valve. Since the sampler lines are vented, disconnecting the cylinder is safely accomplished at atmospheric or sub-atmospheric pressure at the QDCs without contamination to the operator or the environment. After the cylinder is removed the QDC flexible hose that is connected to the top of the sample cylinder can be inserted into the fixed, bottom QDC since there is a male on the flex hose end and a female on the fixed end. See note above.

Volume reduction of the sample - The sample cylinder assembly is now removed from the sampler and excess sample is manually discharged out of the assembly to meet the maximum fill density as noted previously. This procedure is as noted previously and emits VOCs to the local atmosphere and in the area of the operator.

Advantages	Disadvantages
• Allows for the flushing old product from the sampler to increase sample accuracy without wasting product or discharge to the atmosphere • Improves safety by venting of the sampler lines to a reduced pressure and disposing of the vented product to a local or centralized recovery or flare system. • If multi-ported/multi function valves are used (as shown in this Sentry example), the ease of use for the operator is enhanced and maintenance points eliminated.	• Requires an additional step to manually reduce the volume in the cylinder lengthening and complicating the sampling process. • Requires local manual discharge of excess liquid in the sample cylinder assembly to meet maximum fill density. This presents operator safety and environmental concerns and reduces accuracy of the sample since the process is not easily repeated.

2. Speed loop using a sample cylinder assembly and an expansion vessel

   This method provides the product flow advantage of a speed loop with the added ability to provide the maximum fill density on line prior to removing the sample cylinder assembly by adding an expansion vessel and vent valve. Its typical operation (shown with the use of a Sentry Tandem Switching Valve) is shown in figure 2. **Note: Other versions use multiple valves to accomplish the same result and therefore add complexity and maintenance.**

Legend:

1 = Sample inlet connection
2 = Sample outlet connection
3 = Vent connection

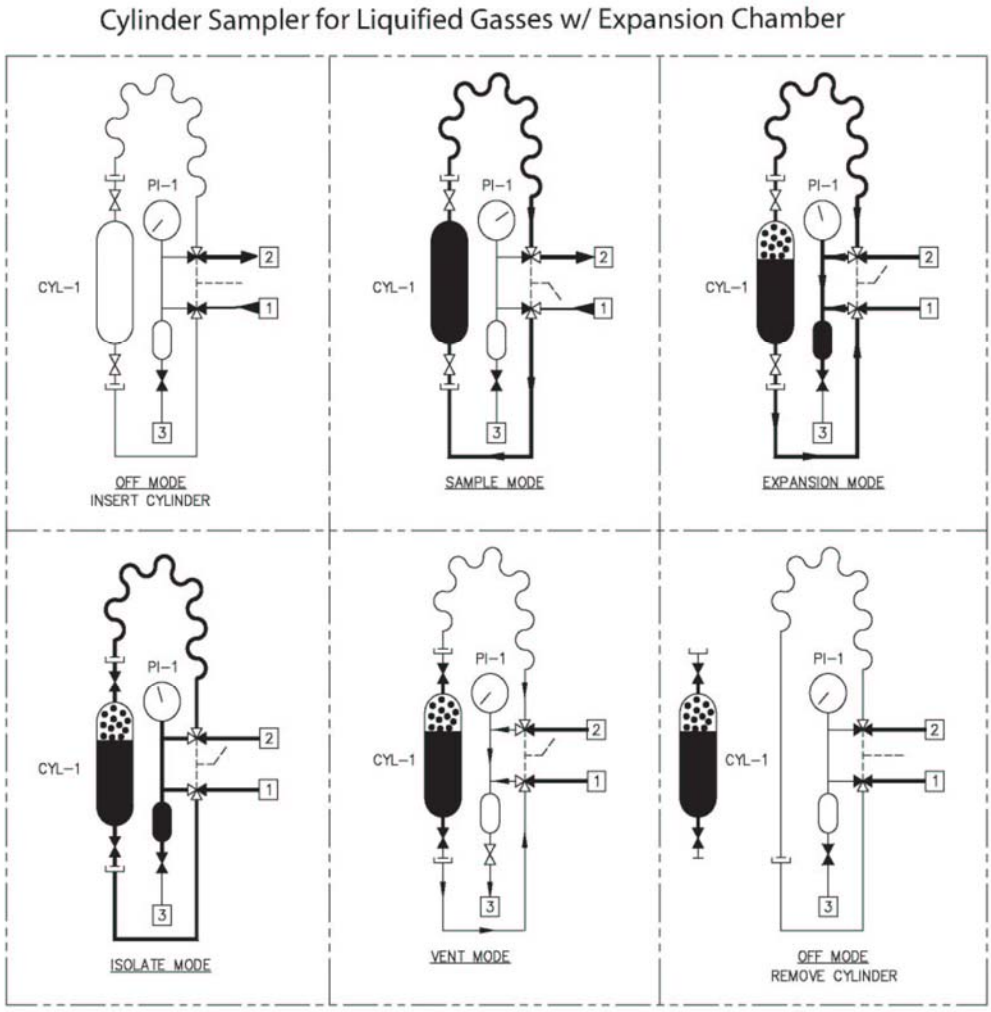

Figure 2

Off Mode (insert cylinder) - The tandem switching valve handle is turned to the off position blocking both side ports on both 3-way valves on the switching valve. There is no flow through the sampler. An empty sample cylinder assembly is placed into the sampler in this mode in preparation for sampling. **Note: Sentry design samplers also use a male and female Quick Disconnect (QDC) set on the sampler (also required on the sample cylinder) so that when the sample cylinder is not on the sampler, the fixed QDC and the hose QDC can be connected to close the sample loop on the unit. This prevents damage to the QDC ends and keeps them clean and minimizes the number of sample cylinders needed for the sampling process.**

Sample mode - The tandem switching valve handle is turned to the sample position and sample now flows through the sampler and the sample cylinder assembly. This is essentially a filling/ purging process. Notice that on LPG (and liquids) the sample inlet to the cylinder is on the bottom. This allows the liquids to push out any old sample or air to the sample return. The pressure gage shows that system pressure is on the sampler. It does not directly indicate sample flow, but provides an indirect indication that sample has reached this point.

Expansion mode - The tandem switching valve handle is turned to the expansion position. This blocks the sample inlet and outlet lines and connects the sample cylinder to an expansion vessel. This expansion vessel is at vent/flare or atmospheric pressure and allows 20% of the sample in the cylinder assembly to expand to a gas and provides for the maximum fill density in the sample cylinder assembly (other maximum fill density levels are available). The pressure gage helps validate that the sample pressure has been lowered in the expansion mode. This process is highly repeatable from sample to sample and operator-to-operator so sample accuracy is greatly improved.

Isolate mode - The valves on the sample cylinder assembly are closed isolating the sample with the outage space in the assembly to achieve the maximum fill density.

Vent mode - The vent valve below the expansion chamber is now opened and the vapors/liquids in the sampler lines and the expansion chamber flash to the vent/flare header or local vapor recovery device. This process is very efficient in eliminating these gases/liquids in the sampler lines for LPG since the vapor pressure of LPG is substantially above atmospheric pressure.

Off mode (remove cylinder) - The switching valve handle is turned to the off position blocking both side ports on both 3-way valves on the tandem-switching valve. Since the sampler lines are vented, disconnecting the cylinder is safely accomplished at atmospheric or sub-atmospheric pressure at the QDCs without contamination to the operator or the environment. The vent valve is closed. After the cylinder is removed the QDC flexible hose that is connected to the top of the sample cylinder can be inserted into the fixed, bottom QDC since there is a male on the flex hose end and a female on the fixed end. See note above. Since the maximum fill density has been achieved while the sample cylinder assembly is connected to the sampler there is no need to manually reduce the volume in the sample cylinder assembly after it has been removed from the sampler. This greatly increases operator safety, improves sample repeatability and eliminates any release of VOCs to the atmosphere.

Other comments - Note that the vent connection 3 is now located downward. This assists in "draining" any liquid from the expansion chamber.

Advantages	Disadvantages
• Allows for the flushing old product from the sampler to increase sample accuracy without wasting product or discharge to the atmosphere • Improves safety by venting of the sampler lines to a reduced pressure and disposing of the vented product to a local or centralized recovery or flare system. • If multi-ported/multi function valves are used (as shown in this Sentry example), the ease of use for the operator is enhanced and maintenance points eliminated. • Maximum fill density is achieved while the sample cylinder assembly is connected to the sampler. It also achieves the same result by a fixed volume every time. This increases sample accuracy and repeatability, improves safety and eliminates discharge of VOCs to the atmosphere.	• Slightly more expensive than the outage tube method.

3. Speed loop using a sample cylinder assembly and a sight glass

Bypass Mode- Four-way valve in bypass mode. Product circulates through the valve from the inlet to the return.

Sample mode - Four way valve in sample mode. Product is circulated from the inlet line through a four-way valve, the sample cylinder assembly and the sight glass back to the return line. Note: The sight glass must be positioned on the sampler to allow for the mid point of the sight glass to be equal to the maximum fill density of the sample cylinder assembly. See additional information below. Product is circulated to flush the sampler and the sample cylinder assembly as appropriate. Flow can be seen in the sight glass.

Capture sample - Four-way valve in the bypass mode. Inlet and return are connected isolating the sampler with product in the sample cylinder assembly, sampler lines and sight glass.

Vent liquid for maximum fill density - Open vent valve and visually see liquid level drop in the sight glass. Liquid level in the mid point of sight glass corresponds to specified maximum fill density. Close vent valve at mid point.

Isolate Sample - Isolation valves on the sample cylinder assembly are closed to capture sample in the sample cylinder assembly.

Vent sampler lines - Open vent valve to reduce pressure in sample lines prior to disconnecting the Quick disconnects to remove cylinder.

Advantages	Disadvantages
• Allows for the flushing old product from the sampler to increase sample accuracy without wasting product or discharge to the atmosphere • Improves safety by venting of the sampler lines to a reduced pressure and disposing of the vented product to a local or centralized recovery or flare system. • View of sample flow through sight glass during sampling process	• Requires two separate steps with different valves to create maximum fill density and vent the sampler. • Visual validation and manual control of the vent valve are required to achieve maximum fill density limiting the repeatability of the sampling process. • Sight glass requires added maintenance. • No off position on the four way valve. Isolation of the sampler requires addition of another valve(s).

B. Single point sampling connection with venting provision

This is a common sampling technique for LPG and other high vapor pressure liquids when there is no possibility of a speed loop because of product sample location in the process. It allows for purging the sampler to the flare/vent header and for maximum fill density to be created while the sample cylinder is still on the sampler. However, product is lost to the flare/vent header during sampler purging. The same methods as shown above for speed loop sample connections are used except that the sample return and the flare/vent connection become the same and a throttling valve is placed in this line to put a back pressure on the line so the sample cylinder assembly can fill with liquid. The Sentry method of using an expansion vessel is show below for illustration. No other examples are show since they are so similar to the speed loop methods. Advantages and disadvantages of each method are also similar.

1. <u>Single point connection with venting using a sample cylinder assembly and an expansion vessel</u>

   When there is no sample return line, the LPG liquid will vaporize as it passes through the sample cylinder assembly and the cylinder will never fill fully with liquid. By adding a valve in the sample outlet line to the flare/vent connection, inlet pressure can fill the cylinder when this valve is closed.

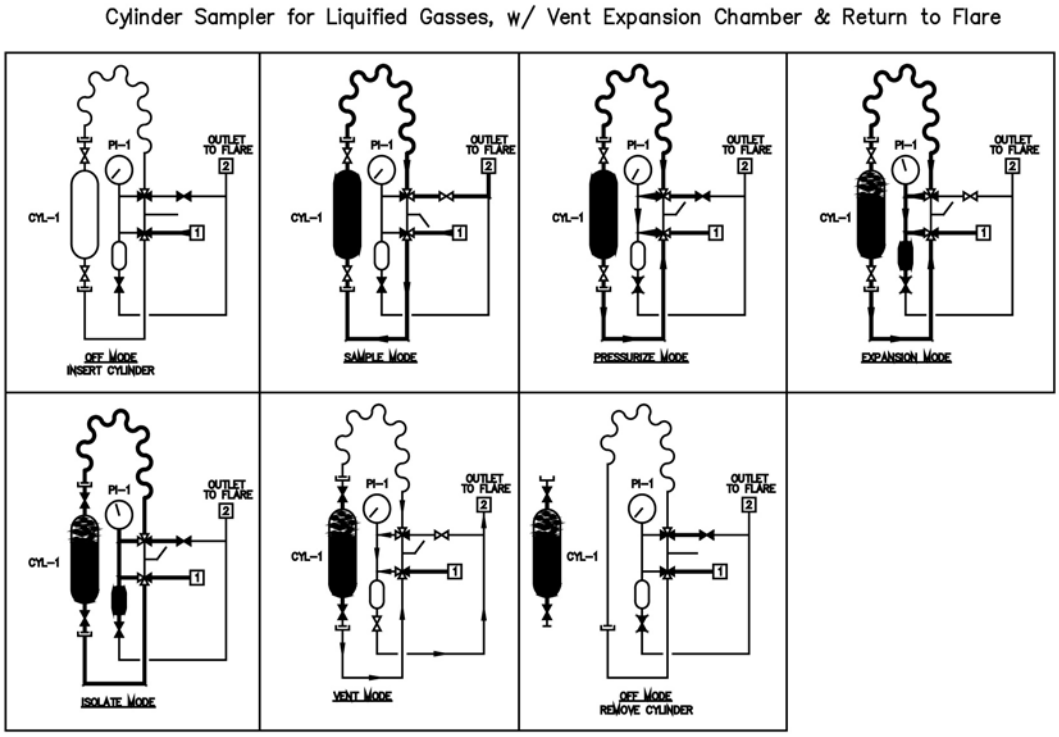

Figure 3

Off Mode (insert cylinder) – The tandem switching valve handle is turned to the off position blocking both side ports on both 3-way valves on the switching valve. There is no flow through the sampler. An empty sample cylinder assembly is placed into the sampler in this mode in preparation for sampling. **Note: Sentry samplers also use a male and female Quick Disconnect (QDC) set on the sampler (also required on the sample cylinder) so that when the sample cylinder is not on the sampler, the fixed QDC and the hose QDC can be connected to close the sample loop on the unit. This prevents damage to the QDC ends and keeps them clean and minimizes the number of sample cylinders needed for the sampling process.**

Sample mode – The tandem switching valve handle is turned to the sample position and sample now flows through the sampler and the sample cylinder assembly. This is essentially a filling/ purging process. Notice that on LPG (and liquids) the sample inlet to the cylinder is on the bottom. This allows the liquids to push out any old sample or air to the sample return. The pressure gage shows a lower pressure than system pressure since the outlet valve is not closed and the discharge is to the flare/vent connection. It does not directly indicate sample flow, but provides an indirect indication that sample has reached this point.

Pressure mode - The throttling valve in the flare/vent outlet line is closed and liquid sample fills the sample cylinder assembly.

Expansion mode - The tandem switching valve handle is turned to the expansion position. This blocks the sample inlet and outlet lines and connects the sample

cylinder to an expansion vessel. This expansion vessel is at vent/flare or atmospheric pressure and allows 20% of the sample in the cylinder assembly to expand to a gas and provides for the maximum fill density in the sample cylinder assembly (other maximum fill density levels are available). The pressure gage helps validate that the sample pressure has been lowered in the expansion mode. This process is highly repeatable from sample to sample and operator-to-operator so sample accuracy is greatly improved.

Isolate mode – The valves on the sample cylinder assembly are closed isolating the sample with the outage space in the assembly to achieve the maximum fill density.

Vent mode - The vent valve below the expansion chamber and the throttling valve in the flare/vent outlet line are now opened and the vapors/liquids in the sampler lines and the expansion chamber flash to the vent/flare header or local vapor recovery device. This process is very efficient in eliminating these gases/liquids in the sampler lines for LPG since the vapor pressure of LPG is substantially above atmospheric pressure.

Off mode (remove cylinder) - The switching valve handle is turned to the off position blocking both side ports on both 3-way valves on the tandem-switching valve. Since the sampler lines are vented, disconnecting the cylinder is safely accomplished at atmospheric or sub-atmospheric pressure at the QDCs without contamination to the operator or the environment. The vent valve is closed. After the cylinder is removed the QDC flexible hose that is connected to the top of the sample cylinder can be inserted into the fixed, bottom QDC since there is a male on the flex hose end and a female on the fixed end. See note above. Since the maximum fill density has been achieved while the sample cylinder assembly is connected to the sampler there is no need to manually reduce the volume in the sample cylinder assembly after it has been removed from the sampler. This greatly increases operator safety, improves sample repeatability and eliminates any release of VOCs to the atmosphere.

C. Cylinder Sampling from single point sample connections without venting

In its simplest form a sample cylinder assembly can be connected to a product line with an isolation valve and vent valve. See figure 4. This type of sampling is not used in many countries due to regulations controlling the release of VOCs and safety regulations. Many other countries are currently implementing similar regulations and operators universally do not like this sampling technique due to the safety concerns.

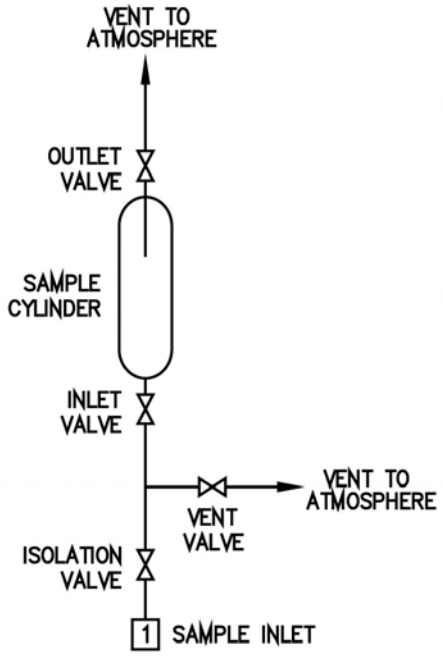

Figure 4

If the sample cylinder assembly has some possibility of previous contamination or used on a different product, it requires purging prior to taking the sample. The sample cylinder assembly is “cleaned” by repetitive filling and discharging product through it and/or by rotating the cylinder 180 degrees and then purging so that the outage tube does not prohibit evacuation of all the gases in the cylinder. The contents of the sample cylinder assembly are therefore discharged to the atmosphere near the point of sampling several times.

After the sample cylinder assembly is purged, product is sampled by closing the outlet valve on the sample cylinder assembly and opening the isolation vent valve allowing the product to fill the assembly through the product isolation valve and the cylinder assembly inlet valve. The sample cylinder assembly inlet valve and the isolation valve are closed. The vent valve is opened to reduce the inlet pressure in the sample line between the isolation valve and the sample cylinder assembly prior to removing it. The sample cylinder assembly can now be removed and the manual evacuation procedure (see outage tube definition) performed to meet the maximum fill density.

Advantages	Disadvantages
• Low cost	• Opening and closing of valves must be in sequence to avoid loss of sample and maintain operator safety. • Significant loss of product and discharge of VOCs to the atmosphere. • Requires an additional step to manually reduce the volume in the cylinder lengthening and complicating the sampling process. Requires local manual discharge of excess liquid in the sample cylinder assembly to meet maximum fill density. This present operator safety and environmental concerns and reduces accuracy of the sample since the process is not very repeatable.

In summary, we believe this sampling method is unsafe, not accurate or repeatable and presents an environmental hazard.

❖ **Frequently Asked Questions**

➢ **Why does Sentry use a tandem switching valve?** Because it simplifies operation and sampling by replacing manual manipulation of multiple valves with two 3 way valves that perform multiple functions at one time. It increases accuracy and is safer for the operator. From an ergonomic perspective, the valve's large handle and wide operating arc make it is easier to turn and reduces operator effort in the sampling process

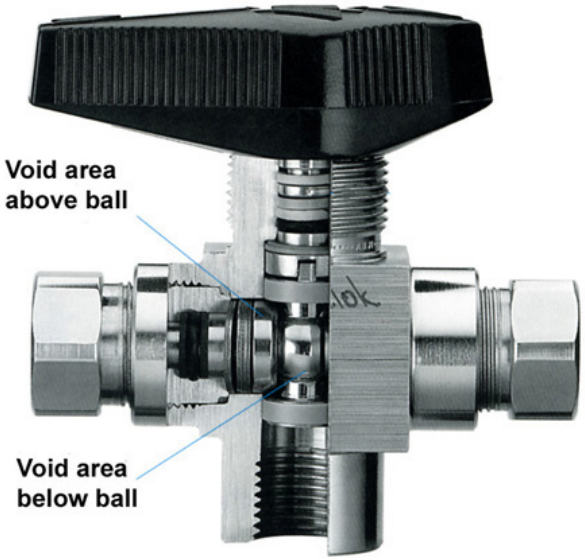

Figure 5

- **What about seals?** Our valves uses a PFA encapsulated ball as noted above and PTFE stem seals. These have good chemical resistance across a broad range of applications. Competitive switching valves have a variety of PTFE seat seals, retaining springs, O-rings, ball bearings and stem seals to accomplish the same result. The valves are more complicated and expensive to maintain.

- **Can we fully automate this process?** Yes, but we don't see the likely use of that in cylinder type samplers for gases and LPG. It would require a compressed gas to drive pneumatic operators and there is usually not a reason to have compressed gas available at the sample station location.

❖ Options for LPG sampler (Sentry Model MLG)

- **Sample cooler** - Cooler option should always include a temperature indicator (TI), throttling sample valve and cooling water valves. We suggest the use of a sample cooler if the sample temperature exceeds 140°F (60°C)

- **Sight Glass/Flow Indicator** – If a positive indication of flow is required, Sentry can add a sight glass. However, we prefer to still use our expansion vessel method of sampling since we think it is better.

- **Block valves** - add inlet and outlet block valves.

- **Enclosure** - These come in all shapes and sizes. Additional sub options include: Materials of construction (stainless, FRP, painted carbon steel, etc.) steam heat, electric heat, insulation, window in the door, etc.

- **Pipe stand** - This is a mounting stand to make the sampler freestanding. All our LPG samplers come standard with the ability to be pipe mounted and this is the preferred method. Consult factory for wind load and other mechanical design details.

- **Purge** – When product being sampled is very hazardous or causes other problems with the sampler, purging configurations with an inert gas is available.

- **Carbon canister on vent** - for local vapor recovery

- **Higher-pressure ratings** - The determining pressure rating factor of the cylinder sampler is usually the sample cylinder. These are normally rated at 1800 psig and this becomes the rating of the sampler. If we make special cylinders, we can rate these at higher pressures.

- **Special Sampler materials** – Glass-lined and Monel® are easily available.

- **Cylinder materials** - PTFE lined cylinder, Monel® cylinder, glass-lined cylinder.

- **Inlet Filter** - Selection is based on particles in the sample that might clog downstream components.

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