TECHNIQUES OF NATURAL GAS SPOT SAMPLING

Adam Johnson
Outside Sales

Welker, Inc.
P. O. Box 138
Sugar Land, Texas 77487-0138 USA

PURPOSE

Collecting a representative sample of natural gas is important for several reasons, but arguably, the main goal of a sampling procedure will be to determine ultimately the value of the gas in question. Other important information that will be gained by analyzing a sample can include specific gravity, total gas composition, or the gas's hydrocarbon dew point level. Gas samples can be gathered by three primary techniques; spot sampling, continuous composite sampling, or continuous on-line analysis sampling systems. This paper is focused on examining the various spot sampling techniques that are currently available in the industry along with their proper implementation, in addition to the most suitable equipment to utilize.

INTRODUCTION

The quantity or volume of natural gas that is produced, transported, stored, and distributed in today's market is ever increasing, and with it the importance of accurate measurement continues to grow exponentially. Verifying the exact composition of the product is not only important from an economic standpoint, but it is also important from a product treatment standpoint as well. The reason being is that natural gas is used as feedstock for numerous petrochemical product streams. The final product, which will be derived from the gas feedstock, depends greatly on the composition and BTU level of the initial gas supply. Any small up front investment that is made to determine correctly the gas composition, will quickly allow one to recoup that investment, time and effort put into the proper equipment, which is designed specifically to obtain an optimum gas sample. Additionally, if approved sampling procedures are followed, the potential for financial disputes between a supplier and customer will be greatly reduced. As natural gas becomes increasingly more scarce and costly to produce, the importance of properly and accurately determining the composition of the gas product will continue to be paramount to all parties involved.

Quoting from the Gas Processors Association (GPA) publication entitled GPA 2166-86, "The object of any sampling procedure is to obtain a representative sample of hydrocarbon from the system under investigation. Any subsequent analysis of the sample, regardless of the test, is inaccurate unless a representative sample is obtained." The International Standards Organization (ISO) standard ISO-10715 describes a representative sample as "A sample having the same composition as the material sampled, when the latter is considered as a homogeneous whole." Citing the American Petroleum Institute (API) Manual of Petroleum Measurement Standards (MPMS), Chapter 14.1, we find a similar statement in the latest revision, "a representative sample is compositionally identical, or as near to identical as possible, to the sample source stream," which is also quoted in the American Society for Testing and Materials (ASTM) International standard ASTM 5287-97. These published standards are the most common reference on accepted natural gas sampling procedures, along with the American Gas Association (AGA) Gas Measurement Manual, Part No. 11, Section 11.3.

Proper and accurate sampling is the bedrock upon which the correct determination of the gas composition is built and the gas sample is, also, the primary source to conclude the specific gravity for the gas being sampled. Specific gravity is a critical component in determining the flow formula, from which the product quantity will be decided. Any error in the sampling procedure will affect both the gas quality and quantity, which ultimately impacts profitability.

GAS SAMPLING

Natural gas sampling has been performed in various guises dating back to the genesis of man's discovery of natural gas vents in China, which were ignited and used to evaporate seawater to collect salt over 2000 years ago. Sampling techniques were refined and passed down from generation to generation as the different uses for natural gas continued to grow. The majority of sampling methods practiced through history are insufficient when measured against today's tighter demands of accuracy and repeatability. However, industry standards have been developed and distributed to reach toward these requirements. Over the last few decades, several sampling standards have been created and published, with the two most widely known being GPA-2166-86 and ISO-10715. The API has revised its gas sampling standard, MPMS, Chapter 14.1, which was published in June 2001. This updated industry cornerstone has generated a renewed and increased interest in proper

sampling techniques, due largely in part to the substantial quantity of clinical test data produced for each sampling method during the latest revision.

Measurement technicians or related personnel who are responsible for physically taking the spot samples should be made fully aware of the seriousness and overall purpose of their task. They should be completely knowledgeable of the proper spot sampling methods, procedures, and techniques involved. Periodic training and educational updates on new standard revisions is a vital responsibility of their position and these employees must comprehend that properly performing their individual roles will have a measurable financial impact on the profits of their company.

A review of the relative sampling standards and the proper operation and installation of the equipment involved is an important step in the complete, accurate sampling process. It is not possible to overstress the importance of proper maintenance of all related sampling equipment and its impact to the success of any sampling method. Dirty, poorly maintained, or malfunctioning sampling apparatus will most certainly affect the accuracy of the final analysis results and, also, the ultimate profitability of the company's operation.

SAMPLING COMPONENTS

The various components of a typical spot sampling system deserve individual consideration, before the various sampling procedures are discussed.

Valves - Condensation can be created if shut-off/isolation valves present a restriction that causes a pressure drop in the gas. It is very important that no leaks be present in any of the valves installed on the collection cylinder. If a leak occurs, the light ends will be the first to escape, thereby causing the remaining sample to be overrepresented with heavy ends (e.g., C_3, C_4, or above). With the understood importance of obtaining a positive shut off, wisdom and experience dictate using valves with soft seals. To avoid fractionization in the sample that can come from using restrictive valve paths, large orifice valves should also be used.

Filters - The use of a filter or membrane element in spot sampling procedures is rare and should be done with extreme caution. If a filter is to be used, proper selection of the filter's flow capacity and particle size is strongly encouraged. A filter that is too small or does not have a sufficient drip pot capacity for gases that have entrained water/liquid/condensate, is likely to result in the taking of an inaccurate or skewed sample.

Pipework/Tubing - Should be as short in length and as small in diameter as practically possible. This will assist in minimizing the time delay as gas proceeds from the probe at the sample point to the collection cylinder. This will also help maintain the sample's integrity.

Heating Elements - Convincing arguments can be presented with sufficient evidence to show that heating all the components of a spot sampling system can be a prudent part of building a reliable and accurate system. Knowledge of the hydrocarbon dew point of a natural gas stream is a critical issue in obtaining a representative gas sample as keeping the gas in the gaseous phase is critical for an accurate spot sample.

Probes - The recommended placement is at the top of the pipe, reaching into the center one third or at least 200 mm (8 inches) for larger diameter pipes; in an area of minimum turbulence, that is, away from headers, bends, valves, etc. Turbulence will stir up any contaminates that typically reside at the bottom of the pipeline and are therefore not normally a part of the flowing gas stream. If a probe were placed at a point of turbulence, these contaminates will likely be taken into the probe creating a sample that is not truly representative of the gas flowing in the pipeline. The design of the sample probe can vary with tips being beveled, 45°, shrouded, square cut, or of some other configuration. Testing has been performed to determine whether these physical designs had any significant impact in the quality of the sample and these tests have proven that probe shape had no overall impact. The important key to remember is to have the end of the probe placed in the center one third of the pipeline in the correct location (positive velocity/no turbulence) with a proper valve on the outlet.

Sample Cylinders - These are typically used for the collection of gases and light liquid hydrocarbons. Single Cavity or spun cylinders have jokingly been referred to as "sample bombs" as exposure to a heat source, if no relief valve or rupture disc is present, can result in an explosion if the expanding gas causes the pressure to become too high. Sample cylinders come in two forms; one is a single cavity cylinder mentioned above with a valve at each end, and the other version is known as a constant-pressure (CP) or floating-piston sample cylinder, which takes the form of a closed end cylinder with an internal piston. A constant-pressure cylinder is prepared for use by pressurizing one side or end, forcing the internal piston to the sample end. When the spot sample is pulled, the sampled product is then collected and stored at whatever pressure is pre-charged at the back of the piston. Using a CP cylinder allows you to collect a sample at a pressure, which will be above the vapor pressure of the

light ends present in the natural gas. Having the floating-piston at the end of the cylinder eliminates the need for excessive purging of the sample equipment. Another benefit of using a constant-pressure cylinder is seen when using a vacuum in the sample cylinder (which is often destroyed by technicians checking to see if a vacuum is in fact present). Using the water or glycol displacement method is also not necessary as the pre-charged piston replaces the liquid that would have acted much in the same manner. Constant-pressure cylinders help guarantee that the actual sample taken is composed entirely of the gas flowing in the pipeline and will not include any ambient air or other contaminants. The cylinder connection is simple and very straightforward making the overall sampling operation easier for technicians and minimizing the likelihood of an incorrect sample being taken.

As the same can be said for all involved components, sample cylinders should be constructed with a material that is compatible with the gas being sampled. For instance, H_2S (hydrogen sulfide) can be absorbed into the atomic structure of 316 stainless steel. If hydrogen sulfide is present in the gas being sampled, this will necessitate coating the inside of the cylinder with a suitable material, such as Sulfinert, to avoid retained contaminants from carrying over into the sample. The resultant sample will not be truly representative otherwise. Sample cylinders are normally protected with bursting discs which are less expensive and lighter weight than relief valves, though their proper selection and replacement should be a primary concern and not taken lightly. These safety items are used to prevent serious harm, danger, or even death!

Along with the notes on the various sampling components should also go the comment, which is one of the basic rules of sampling: ***The materials of construction of the sampling equipment that come into contact with the sample are to be compatible with the product being sampled.*** It is typically safe to use 316 stainless steel and Viton elastomeric components in the vast majority of sampling situations and one should look for these materials in selecting equipment and ask questions of suppliers about proper material selections if the application requires a unique approach.

LOCATION AND PRODUCT

The sample point should be located in a section of the pipeline that includes a positive velocity, a minimum of turbulence, and the tap mounted on top of the pipe. If this simple guideline is followed, it will effectively eliminate meter manifolds, blow down stacks, standby runs, pig traps, headers of all types, drips, or any type of dead end line.
Also, samples should never be taken adjacent to internal obstructions such as; control valves, orifice plates, elbows, tees, or other fittings that might generate aerosols. Careful attention should also be paid to free liquids (aerosols) in the stream that may be drawn into the sample probe, as subsequent testing on the gas sample cannot account for any liquids contained in the cylinder. Gas Chromatographs cannot accommodate liquids and expensive damage and service time will result if free liquids are not accounted for.

An additional major factor in any correct sampling procedure is an awareness of the hydrocarbon dew point (HCDP) of the gas stream being sampled. The importance of knowing the HCDP is related to (1) the ambient temperature; (2) the temperature of the equipment being used to collect the sample; and (3) the temperature of the flowing stream. The creation of liquids due to equipment design and equipment temperature must be avoided. Determination of the HCDP of the gas stream can be done by the chilled mirror method or by the use of a number of “equation of state” models for hydrocarbon dew point determination. There are several programs available such as Peng-Robinson or SRK. The variations of the calculated results between different equations of state can vary enough that it is strongly recommended to add 20° to 50°F (11° to 28°C) to the answers found. This is to assure the operator that he or she is designing a sampling system that keeps the temperature requirements above the actual hydrocarbon dew point of the sampled stream.

SPOT SAMPLING

While there are several supported or accepted techniques for spot sampling of natural gas, the two most common methods in use today are the fill-and-purge method detailed in GPA-2166-86, Section 7.1 and the constant-pressure or floating-piston cylinder method detailed in Section 7.7.

Spot sampling was the primary method of acquiring a sample for analysis until the early 1970s and is still a widely used method today in a variety of settings. The industry’s current environment which is focused on growing trends toward therm-measurement and therm-billing (BTU values), spot sampling is becoming increasingly more expensive due to the analytical cost and man-hours involved, as well as a questionable method of assessing an accurate heating value to large cubic foot volume sales. It is, quite literally a "spot" sample of what was present in the pipeline at that moment the sample was taken. Anything present in the pipeline at any other time

before or after the spot sample was collected can be no more than a guess. While this level of information may be a reasonable risk if the source of the gas is well known, demonstrated by a lengthy historical data base, most gas being consumed today is a combined product assembled from several origins or wells, or it is switched from source to source by contractual updates; in some cases by daily or even hourly arrangements. Scientific and practical experience has demonstrated that as a natural gas well ages, its gas production grows increasingly richer as the heavier hydrocarbon components, carrying a higher BTU value, naturally take longer to escape the well. The longer the well is in production, the higher the BTU value will tend to become, which correlates to a more valuable product stream. By nature, natural gas is an extremely fragile entity and almost every step in the production, transportation, and distribution of natural gas can or will have an adverse effect on its overall quality. Switching from well to well, pipeline pressure changes, ambient or pipeline temperature changes and various storage methods are only a few of the influencing factors that can add or subtract important BTU values to the gas moving through a measurement station. Given this nearly constant variation in a gas supply chain, it is very possible a spot sample may not represent the correct gas source in question.

In the industry's early years spot sampling was performed in such a manner that the gas was directly introduced into the cylinder by either a probe or connecting the cylinder directly to the pipeline until pipeline pressure was reached. The cylinder was then closed, removed from the pipeline and shipped to the laboratory for calorimeter or gas chromatograph analysis. As the need to know the heating value/quality of the gas (BTU value) became increasingly more important, versus simple quantity measurement, tests were conducted to determine if the natural gas was being altered by the sampling procedure used. Once the results were examined, it was apparent contaminates such as atmospheric air were being introduced to the collected sample and it became clear that a new filling method was therefore needed. The Fill and Purge method was thus adopted and after a period of time it was determined that retrograde condensation was occurring by this sampling process and thus an even newer spot sample method was created. This newer method is now known as the **GPA** method using a spot sample manifold and pigtail extension for filling the standard or single-cavity cylinder. This **GPA** method reduced the negative effects found with the "filling only" procedure. The manifold allows the gas to be "trapped" in the cylinder at full pipeline pressure, rather than simply "dead ended" into the cylinder (i.e., zero pressure up to line pressure). In addition, it was discovered sampling situations with free liquids present in the pipeline required a special approach and the **GPA** liquid separator was developed and added to certain techniques.

As the overall quality of the natural gas became a highly critical component of the billing process, along with volume (std. cu. m. or std cu. ft.), the industry again reviewed the single-cavity cylinder and its effects on sampling accuracy.

It became very evident that keeping the gas at pipeline pressure from start to finish in the sampling process was highly important to overall accuracy. Any reduction in pressure or a change in temperature from the pipeline condition at the time of sampling was found to alter the gas analysis in almost every case examined. Only low BTU gas (975 BTU and below) seemed to escape any noticeable alteration.

Testing and practical observation revealed that when the single-cavity cylinder was being filled, the heavy ends present in the gas sample (e.g., propane, butane, iso-butane, pentane, etc.) dropped out as liquid condensate inside the cylinder until higher pressures were reached. Adopting and implementing the **GPA** spot sampling method helped eliminate this problem, but when the gas sample was being bled out of the single-cavity cylinder into the GC (gas chromatograph), it became evident there was no way to keep the pressure elevated inside the cylinder. As the single-cavity cylinder was opened, the light ends present in the gas sample tended to escape first (e.g., methane, ethane, etc), thus giving a certain, but not completely accurate, BTU value upon analysis. As the lab analysis continued, the sample's BTU value would steadily increase due to the heavy ends remaining inside the cylinder eventually escaping, thus altering the final BTU value compared to the first analysis. Typical custody transfer situations call for more than one test to be performed on any given sample due to concerns of valuation accuracy. Repeatability therefore was more often than not impossible as the more a sample was analyzed, the more the BTU value increased. It became clear that the decrease in pressure inside the cylinder altered the gas composition and yielded a non-representative sample.

The constant-pressure or floating-piston cylinder was therefore developed as an answer to this problem. With an internal sealed floating piston, it became possible to pressurize (pre-charge) the cylinder with an inert gas supply (or the pipeline gas itself) and then reverse the cylinder and fill it slowly from the opposite end. Allowing the gas to push against the piston while "slowly" venting the pre-charge gas, the sample was able to be taken at full line pressure from start to finish. This

resulted in a cylinder containing a gas sample that accurately reflected the true pipeline conditions for pressure and phase. Once the sample reached the laboratory, an inert gas supply could be connected to the pre-charge (supply) side of the cylinder equal to the pipeline pressure. The pre-charge gas then forced the floating piston to the opposite product end, injecting the gas sample into the GC. While the cylinder is being emptied, full pipeline pressure is being constantly maintained and the sample gas composition is not being altered as a result of any pressure reduction. The cylinder can be stored or sent to another laboratory for results confirmation, and when the remaining gas is analyzed, it will again give repeatable results because the pipeline condition of the gas sample is constantly maintained by the constant-pressure cylinder.

One look at a constant-pressure cylinder will quickly reveal it is far different from a single-cavity cylinder. CP cylinders are equipped with positive shut-off valves, safety reliefs, or burst discs and gauges on both ends. This configuration allows the pressure to be controlled and monitored at all times on both ends of the cylinder. The temperature would also be maintained just as with single-cavity cylinders, i.e., heating blankets, ovens, or water baths. The use of a CP cylinder has been proven to give extremely accurate results in both spot sampling as well as in automatic or composite sampling systems. Extensive tests have revealed that a CP cylinder can yield consistent analysis results to within a half of a BTU of the pipeline, when compared to an online or laboratory GC. Employing a constant-pressure cylinder in sampling methods will consistently perform at this high level and the richer, or higher the BTU level of the gas, the more alteration occurs with older (single-cavity) methods.

The CP cylinder also brings with it additional health and environmental safety advantages in handling the gas sample. Purging the cylinder and venting large amounts of natural gas to the atmosphere can be done away with, as all that is needed is a brief purge of the sample line up to the cylinder before the cylinder is filled. The piston is at the sample (product) end of the cylinder when you begin collecting the sample so there is no "dead volume" to purge out to the atmosphere. This saves not only time, but also money as far less gas is wasted into thin air.

Due to the design of the CP cylinder, with seals on the end of each cap, it cannot explode due to over filling. If the cylinder is ever subjected to excess pressure, the safety reliefs will fail, allowing the pressure to escape. In the rare event that they do not function properly, the cylinder body itself will swell and cause the end cap seals to fail, allowing the pressure to escape to the atmosphere safely.

Constant-pressure cylinders have competently served the natural gas industry for over 30 years. Their correct usage provides increased accuracy and success for improved sampling procedures, better sampling systems, repeatability, safer handling, more accurate analysis, and prolonged storage of gas samples. This also includes the storage and transportation of gas and liquid standards for the laboratory.

Errors in correct measurement are becoming increasingly more costly due to the ever-escalating value of a single BTU. Industry wide, more companies are actively improving their sampling methods, techniques, and procedures and leaving behind history's more outdated spot sampling practices.

All updated ISO, GPA, ASTM, AGA, and API standards and committee reports address the proper usage of standard and constant-pressure cylinders for the gas and liquids industry.

TRANSPORTATION

The safe and correct transportation of natural gas samples is a very important issue for all parties involved and for the individual personnel who are transporting the samples. The United States Department of Transportation (DOT) covers the transportation of hydrocarbon samples in CFR-49. Each party involved in transporting sample cylinders and other sampling apparatus, both to and from sample collection locations, should be thoroughly familiar with the rules and regulations set forth in CFR-49.

As well as the safety issues, markings, and forms that are to be filled out for DOT purposes, other considerations should be addressed as well. Among these are:

- Proper tagging of the cylinder for time, date, and location of the sample.
- Pressure and temperature of the pipeline source.
- Identification of the technician who took the sample.
- Method used to obtain the sample.
- Plugging of the valves and checking for leaks prior to transport.
- Protection of the cylinder and sample apparatus during transport, both to and from the sample location.
- Temperature concerns during transport, both to and from the sample location - if necessary or required.

- Other company procedures that will assist in the success of a quality sample being delivered to the laboratory for an accurate analysis.

CONCLUSION

It is difficult to overstress the importance for everyone involved in the natural gas industry to fully understand and support the methods, techniques, and equipment used for spot sampling. Regardless of the application or installation, there is a technique which best meets your needs, and will effect your company's overall profit and loss. Accurate and correct sampling and metering are the revenue streams of your company. Examine your methods, procedures, and needs closely to be sure you are doing your part to contribute to a successful sampling program.

REFERENCES

"Techniques of Natural Gas Spot Sampling," D. J. Fish, April 2002.

"Determining Accurate Gas Quality" L. Witt.

"Proper Sampling of Light Hydrocarbons," O. Broussard, Oil and Gas Journal, September 1977.

"Standard Cylinder vs. Constant-Pressure Cylinders," D. J. Fish, Gas Industries, January 1994.

"Analyzing Heating Value," T. F. Welker, Pipe Line Industry, October 1990.

"Natural Gas Sampling," T. F. Welker, Presented at AGA Annual Meeting, Anaheim, California, 1981.

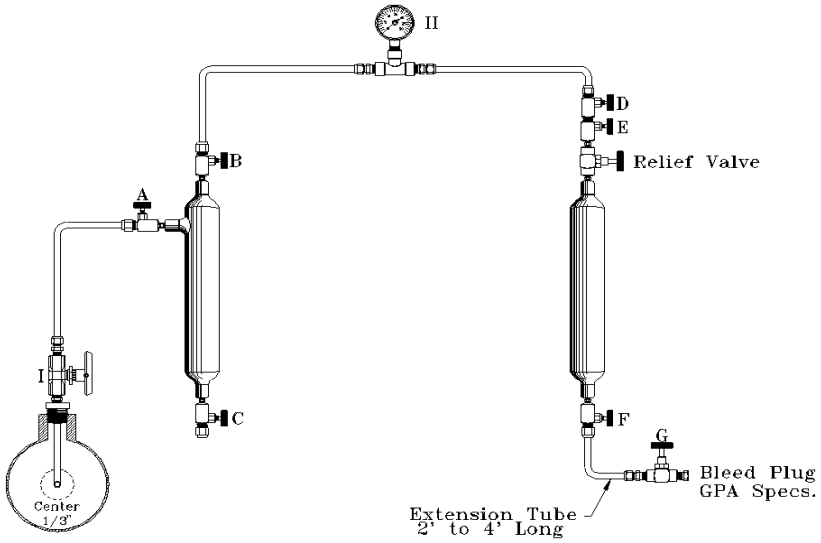
"Methods, Equipment & Installation of Composite Hydrocarbon Sampling Systems," D. J. Fish, Presented at Belgian Institute for Regulation and Automation, Brussels, Belgium, 1993.

"Practical Considerations of Gas Sampling and Gas Sampling Systems," D. J. Fish, Pipeline and Gas Journal, July 1997.

"Selection and Installation of Hydrocarbon Sampling Systems," D. A. Dobbs & D. J. Fish, Presented at Australian International Oil & Gas Conference, Melbourne, Australia, 1991.

Various standards of AGA, GPA, API, ASTM, and ISO.

ATTACHMENT 1



G.P.A. FILL-AND-EMPTY METHOD TYPICAL RECOMMENDED MANIFOLD HOOK-UP

I. Sample probe

II. Sample pressure gauge (same as line pressure)

Steps to follow to draw sample:

1. Open Valve 'I' to blow any accumulated foreign matter in probe or valve.
2. Connect manifold to probe Valve 'I' (close Valve 'I').
3. Open Valves 'A,' 'B,' 'D,' 'E,' and 'F.' Valves 'C' and 'G' remain closed.
4. Open Valve 'I' to allow full pipeline pressure to fill complete manifold.
5. Close Valve 'A' and open Valve 'G' to allow gas in the manifold to bleed to 0 psig.
6. Close Valve 'G' and open Valve 'A' to allow pressure to build rapidly to full line pressure.

   NOTE: Steps 4 through 6 should be repeated until all air has been eliminated from the system (3 to 5 times).
7. Open Valve 'A' quickly to fill manifold with full pipeline pressure. Close Valve 'A.'
8. Open extension Valve 'G.' Allow pressure to bleed to 0 psig. Close Valve 'G.'

   NOTE: If at any time liquids appear at Valve 'G,' the sample should be discarded.

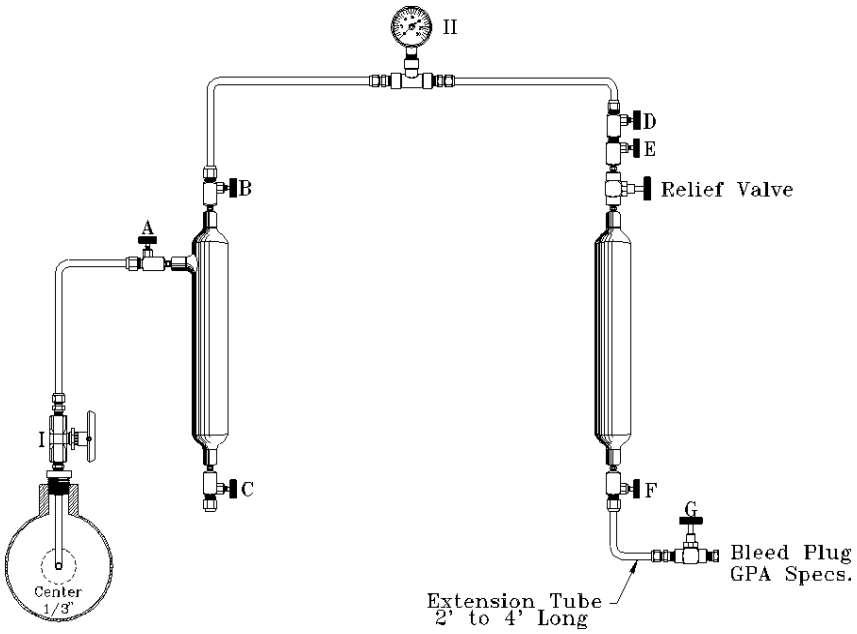
   NOTE: Steps 7 and 8 constitute the fill and empty cycle and should be repeated as many times as required by your pipeline pressure and the chart below. Table 1 gives the minimum number of purges required to condition your sample cylinder (providing the sample cylinder is clean and dry).

TABLE 1

Maximum Gas Pressure (PSIG)	Number of Purge Cycles
15 - 30	13
30 - 60	8
60 - 90	6
90 - 150	5
150 - 500	4
Over 500	3

9. At the completion of the proper number of purge cycles, close Valves 'E', 'F,' and probe Valve 'I.' Bleed the pressure from the manifold and extension tube. Remove the sample cylinder from the manifold and check for leaks. Plug the valves. Fill out the proper sample tag and put the cylinder in a proper carrying case to be transported to the lab.

ATTACHMENT 2



G.P.A. CONTINUOUS PURGE METHOD TYPICAL MANIFOLD HOOK-UP

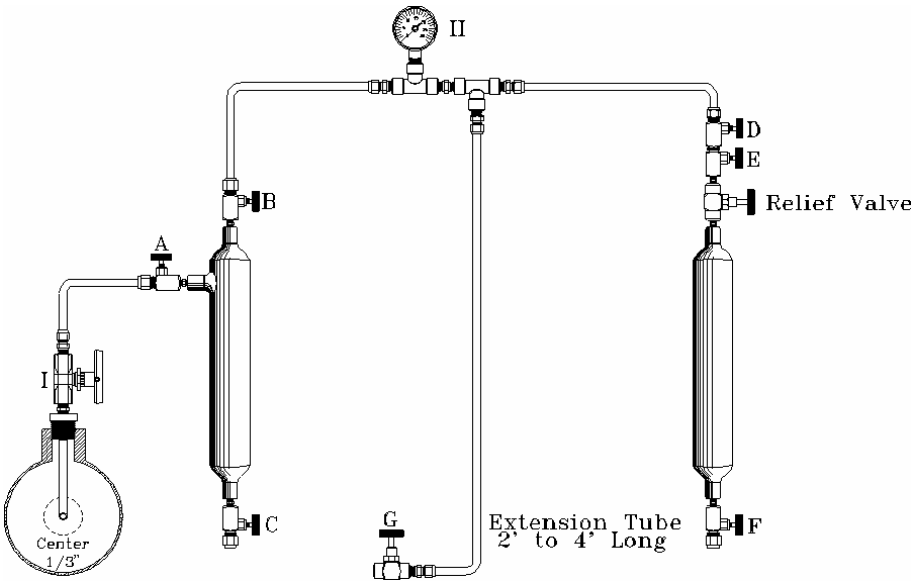
Caution: This method should not be used on wet natural gas or gas over 400 psi.

I. Sample probe
II. Sample pressure gauge (line pressure)

Steps to follow to draw a sample:

1. Open Valve 'I' to blow out any accumulated foreign matter in probe or valve (close Valve 'I').
2. Connect sample manifold to probe Valve 'I.' All valves closed.
3. Slowly open Valve 'I,' and then slowly open in sequence Valves 'A,' 'B,' 'D,' 'E,' 'F,' and 'G.'
   NOTE: Valve 'G' must have flow plug installed.
4. Allow gas to flow through the manifold for a minimum of 3 minutes.
   NOTE: That more time will be required if the sample cylinder has not been properly cleaned and dried.
   NOTE: The G.P.A. method should be consulted for unusual conditions.
5. After specified time, valves should be closed beginning with Valve 'G' and working toward the source valve, sequence 'G,' 'F,' 'E,' 'D,' 'B,' 'A,' and 'I.'
6. Bleed pressure from manifold and remove sample cylinder.
7. Check cylinder for leaks and plug valves.
8. Fill out sample card and put cylinders in proper box for shipment.

ATTACHMENT 3



G.P.A. METHOD FOR TAKING A SPOT SAMPLE IN AN EVACUATED CYLINDER OR STANDARD SAMPLE CYLINDER FILLED WITH AN INERT GAS

1. Evacuate a clean sample cylinder and plug valves.
2. Open probe Valve 'I' to clean valve and probe of accumulated materials.
3. Connect manifold to probe Valve 'I.'
4. With all valves closed, attach sample cylinder to manifold.

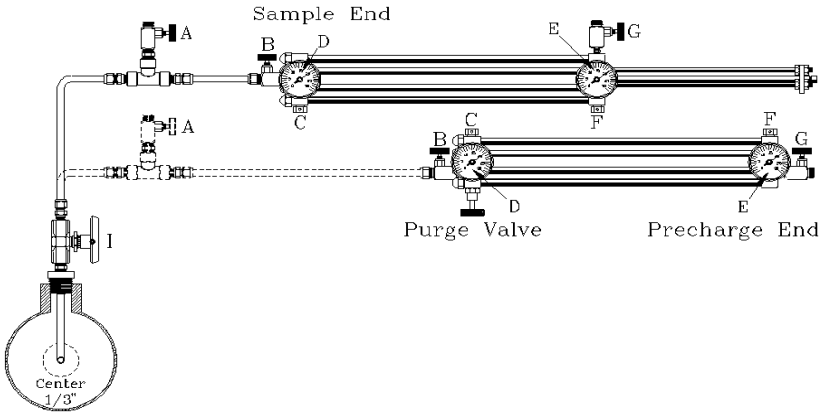
   NOTE: Check with a gauge to ensure sample cylinder is evacuated.
5. Open probe Valve 'I,' 'A,' and 'B.' All other valves closed.

   NOTE: Sample gauge II will indicate pipeline pressure.
6. Close Valve 'A.'
7. Open Valve 'G' on the extension tube.

   NOTE: This will bleed manifold pressure to 0 psi.

   NOTE: Steps 6 and 7 constitute a purge of the piping to the evacuated cylinder. This should be repeated as required by your pipeline pressure to ensure any air in the lines has been purged with gas.
8. Open Valve 'A' with Valve 'B' open, slowly open Valve 'D' and allow line pressure to fill sample cylinder.
9. Close Valve 'D.' Close Valve 'I.' Open Valve 'G' to remove pressure from the sample manifold.
10. Remove sample cylinder from manifold. Check for leaks, plug valves, fill out sample information, and put cylinder in an appropriate case for transportation.

ATTACHMENT 4



DRAWING A SPOT SAMPLE INTO A CONSTANT-PRESSURE (FLOATING-PISTON) SAMPLE CYLINDER

To draw a spot sample into a constant-pressure sample cylinder, the following procedure should be used.

Prior to going to the field, check these items:

1. Has the cylinder been emptied, cleaned, and checked for leaks?
2. Attach to the pre-charge Valve 'G,' a supply of inert gas (N_2), with Valve 'B' open, and fill the cylinder to 100 psi above line pressure. Close Valves 'G' and 'B.'
3. Put the cylinder in a carrying case with the proper transport papers, and go to the field.

At the sample point:

1. With an appropriate connector, connect Valve 'B' on the cylinder to the process connection 'I' (sample probe).
2. Valves 'I,' 'A,' 'B,' and purge valve are closed.
3. Open Valve 'I.' Check the connections for leaks.
4. Open Valve 'B.' This allows product to flow into the cylinder to the face of the piston.
5. Purge: Close Valve 'I' with 'B' open, open 'A' (slowly). Bleed off the product in the cylinder.
6. Close Valve 'A.'
7. Open Valve 'I.'
8. Close Valve 'I.''
9. Open Valve 'A' (slowly).
10. Close 'A.'

NOTE: By opening 'I' and 'A,' you fill and empty the piping and cylinder to eliminate air or contaminants. If the pressure is above 500 psi, 3 to 5 purges will be adequate. Do not flow through with all valves open. This may cause retrograde condensation to occur.

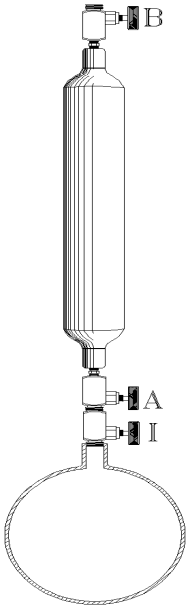
To draw the sample after the purge:

1. Open Valve 'I.'
2. Open Valve 'B.' Valve 'A' remains closed. Product is now against the face of the piston.
3. Slowly open Valve 'G.' Bleeding off the pre-charge from the backside of the piston will allow the process pressure to fill the sample container.
4. When the indicator reaches 80%, close Valve 'G.' You now have 80% product, 20% pre-charge, and the cylinder may be transported.

To disconnect the cylinder from the process:

1. Close Valve 'I' (probe valve) and Valve 'B.'
2. Slowly open Valve 'A' to remove the pressure between the purge valve and cylinder inlet valve 'B.'
3. Remove connection at Valve 'B,' plug all the valves, fill out the paperwork, and put the cylinder in a carrying case for transportation.

ATTACHMENT 5



TYPICAL SAMPLE HOOK-UP OF CYLINDER

NOTE: Not recommended.

Sample taken by simply viewing into a convenient valve is not a proper means by which to take a representative sample of any type.

ATTACHMENT 6