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OBTAINING NATURAL GAS SAMPLES FOR HYDROCARBON OR SULFUR ANALYSIS

SCOPE: The purpose of this paper is to inform our customers, and those technicians who sample Natural Gas or gaseous products in the field, as to the recommended procedures for obtaining these samples. This discussion only covers the method commonly used to obtain Natural Gas and most gaseous products, referred to as the "Trap & Purge" procedure. Understanding that all "accurate" analyses begin with accurate sampling and sample handling after they are obtained, is critical. How a sample is taken, and how it is handled prior to analysis, are essential in an accurate analysis of Hydrocarbons and Sulfur compounds. The Trap and Purge method is considered one of the most widely utilized procedures for sampling Gas products in the field, along pipelines, or in Gas processing facilities.

This procedure is only to be used as a guideline of individuals sampling at pressurized Natural Gas sites. This method does not cover sampling procedures for obtaining atmospheric, or negative pressure, samples. This procedure should only be used for sampling gases above 15 psig. Also, sampling "wet" gases, with BTU values above 1100, should only be performed with a line probe and separator, in accordance to GPA- 2166 methodology. Wet gases have increased amounts of Propane+ hydrocarbons that can condense out in the sampling cylinder , yielding erroneous results. Sampling these gases should only be performed through a sampling manifold or sample separator, which allows condensable (or Free) liquid hydrocarbons to be retained prior to entry in the sampling cylinder. Entrained liquids are the chief cause of obtaining a non-representative, or inaccurate, sample for analysis. A "dry" Natural Gas is defined as one which does not form condensate upon cooling by a rapid expansion from source pressure to atmospheric, or any intermediate pressure.

SECTION 1. EQUIPMENT FOR SAMPLING NATURAL GAS FOR HYDROCARBON AND SULFUR ANALYSIS.

HYDROCARBON ANALYSIS SAMPLING

1) **Sampling Manifold** - A high pressure rated sampling transfer line made of stainless steel, or other flexible material, which is non-reactive to the product being sampled. While Stainless steel is preferred for pressures above 1000 psig, high pressure rubber hoses can be used for obtaining Natural Gas samples. The manifold should have a length of between 1 to 5 feet for flexibility, and a diameter of ¼", or less. It should be equipped with a control valve, a vent valve, and pressure gauge to assure maximum sample cylinder pressure of 1800 psig is not exceeded.

2) **Sampling Cylinders** - Use DOT approved metal cylinders of a type which insures maximum safety, and are corrosion resistant to the product being sampled. For hydrocarbon analysis sampling, it is not necessary to use Teflon lined, or treated steel (Sulfinert) cylinders. Those cylinders are utilized when sampling Natural Gas for Sulfur compounds. Standard stainless steel (304 or 316) or aluminum cylinders, rated to maximum of 1800 psig, should be adequate for natural gas sampling when Sulfur testing is not required. When sampling is completed, all cylinders should have steel end caps placed securely on each valve to prevent loss during transit.

All sampling cylinders should be double valved (one at each end) and pressure tested to 1200 psig prior to use, to assure there are no leaks and that the valves stems seal securely. All cylinders should have a pressure relief disc at one end when sampling gaseous products over 1000 psig.

If the gas source contains a significant amount of Carbon Dioxide (over 10%), or Hydrogen Sulfide (over 1%), the cylinder should either be Teflon lined or treated steel, to prevent reactivity with the metal walls of the cylinder. This reactivity, or passivation, can results in a loss of components during storage, yielding inaccurate results.

All sampling cylinders should be dry and free of any residues from prior sampling.

SULFUR ANALYSIS SAMPLING

1) Sampling Manifold - As discussed above, a high pressure rated, sampling transfer line made of treated stainless steel. This sampling manifold must be either Teflon coated or treated in such a manner as to prevent sulfur passivation during sampling. You should not use standard stainless steel, or rubber, manifolds when sampling natural gas products for Sulfur analyses. Hydrogen Sulfide will react immediately with stainless or non-treated steel surfaces, resulting in lost components through passivation. Always check your cylinders, valves, and manifolds to assure they are coated in some manner as to prevent sulfur absorption.

The sampling manifold should be as short as necessary to obtain the sample from the sampling source. The manifold should also contain a vent valve and shutoff valve which are coated to prevent absorption.

2) Cooling Coils - When sampling natural gas streams for sulfur analysis, it is often necessary to utilize a cooling coil to reduce the temperature of the gas before it enters the cylinder. Quite often, gases from Desulfurization beds are in excess of 500 F, which is hot enough to damage or disable a treated steel surface or Teflon coating, not to mention the Teflon valve seats which can melt above 350 F. The cooling coil should be fashioned from treated (Sulfinert) or glass-lined stainless steel tubing to prevent loss or damage to equipment and sulfur components to be tested. The coil should be made of ¼", or ½" tubing, approximately 30 feet long and coiled to a diameter of roughly 8 to 10 inches. The coil is attached to the high temperature sample point and submerged in a pail of cold water when sampling. Gas from the sample point is slowly moved through the coil, allowed to cool to below 200 F, before entering the sampling cylinder. The coil should include a shutoff valve on the sample source end of the line and also one on the sample cylinder end.

NOTE: These cooling coils can become very hot if the gas moves through them too quickly. The shutoff valves can also be extremely hot. Always use the proper safety gloves and eyewear when sampling these hot gases.

3) Sampling Cylinders - All sample cylinders used for sampling Natural Gas which will be analyzed for Sulfur compounds MUST be either treated stainless steel (Sulfinert brand) or Teflon lined. Sampling cylinders should be pressure rated to 1800 psig and DOT approved for transit.

All cylinders used in sampling natural gas products for Sulfur testing should be subjected to a "passivation" test, prior to use. This procedure requires the analyst or field technician to submit a calibration mixture of various Sulfur compounds to each cylinder to be used. The calibration mixture is allowed to sit (or react) with the cylinder walls for a minimum of 24 hours. At the end of this period, the contents of the cylinder are tested and the remaining Sulfur compounds are compared to the original mixture. If the Hydrogen Sulfide, or any of the Sulfur compounds, are reduced in concentration after 24 hours, the cylinders should not be used for sampling. A "good" treated steel or Teflon lined cylinder will not lose more than 0.5 % in 24 hours, if any at all. Likewise, all cylinders should have adequate valve on both ends which also pass a 24 hour passivation test. These valves are often Teflon coated, or treated steel, which can be damaged by high temperatures from desulfurization treater outlets. Knowledge of the sample points will help prevent these soft seats from damage. Be sure you check the temperature of the gas you are sampling prior to cylinder entry.

4) **Soap Solution & Safety Gear –** A soapy water solution should be available at the time of sampling, to check the cylinder and valves for leaks. A 1% soap in water solution is sprayed around all applicable valves and outlets to assure the cylinders do not leak off during transit. Simply spray a small amount of the solution over the valves and valve ends. If bubbles are visible, then the cylinder valve (or threads) is leaking your sample. Even though valves and cylinders are pressure checked before use, it is possible to have particulate or damaged coatings lodge in the valve stem, causing the cylinder valves to fail when tightened. **Do not attempt to overtighten valves with wrenches to stop leaks.** It has also been observed that high temperature gases (even those which have cooled down in a coil prior to sampling) can melt the soft Teflon valve seats and cause them to leak, despite tightening. If the soap solution bubbles after sampling, replace the cylinder

In addition, always wear the appropriate safety gear, including gloves, eyewear, and FRC clothing, when sampling Natural Gas. It is always possible for sampling cylinders and sample manifolds to rupture during high pressure sampling. Cylinders equipped with pressure relief (rupture) discs can also become a source of release when sampling high pressure gases. Also, FRC (Fire Retardant Clothing) is essential when sampling flammable or explosive gases. Avoid sampling in wool, or synthetic blend clothing materials, as they will build up static electricity and could provide a source of ignition when sampling hydrocarbon gases.

SECTION 2. SAMPLING METHOD FOR NATURAL GAS PRODUCTS – TRAP & PURGE PROCEDURE

1) Prior to sampling, exam the outlet valve of the sample point you wish to sample. Be sure that it is free of particulate, and any condensed or accumulated liquids. If necessary, clean the valve with a steel wire brush to remove dirt, Iron filings, and old Teflon tape. Wipe around threads with clean, dry cloth. Make sure to exam the valve carefully for stress fractures, worn seals, or anything that might impede obtaining a good, clean sample. Old valves can break during sampling and those exposed to H2S environments may be weakened over time. If necessary, replace valve before attaching your sampling hose or manifold.

2) Place a small piece of Teflon tape around the inlet threads of your sampling manifold or hose. Carefully attach your sampling manifold to the sample source. Make sure you record the source pressure before you open the source valve to flow gas. If the pressure is above 1500 psig, or 250 F, do not attempt to obtain the sample. Make the necessary adjustments (ie, cooling coils, partial pressure regulator) before attempting to sample. If the sample source pressure is below 1500 psig and the temperature of the gas is less than 250 F, then you may attach your sample cylinder to the manifold or hose. Make sure you use Teflon tape to seal all threaded connections on your manifold or hose, and cylinders, before sampling.

3) With the sampling cylinder attached to your manifold (all valves should be closed at this point) Carefully open the gas from your sample source, allowing it to fill up the manifold to source pressure. Slowly open the manifold vent valve and allow the source gas to purge through the manifold, venting the effluent to atmosphere. Continue flowing through the manifold until any precipitated liquids have passed. If you continue to visually note intermittent condensed liquids from the sample source through the manifold, you may require a small separator to drop them out. Do not open the cylinder to admit the gas sample until you are sure you have a homogenous, dry gas stream through your manifold. Continue flowing dry gas from your sample source through your manifold for 30 to 60 seconds.
NOTE: Freezing (below 32 F) ambient temperatures outside during sampling can cause hot or warm process and treater gases to cool rapidly, causing condensable hydrocarbons to fallout in your manifold.

These droplets of hydrocarbon liquids must be removed before sampling. If you continue to notice moisture or hydrocarbon droplets coming through your manifold, you may need to install a separator ahead of the manifold. Historical data of the sample source may yield a Dew Point of the gas stream above the ambient temperatures outside. If this occurs, then the condensable components of the gas stream may continue to form droplets while sampling, yielding inaccurate results.

4) After purging sample gas from the source through the manifold, close the vent valve and allow the manifold to fill to source (line) pressure. Carefully open the sample cylinder valve attached to the manifold and slowly allow the cylinder to fill up with sample until it equilibrates to line pressure. Close the valve to the sample cylinder, trapping the sample gas inside. Slowly open the outlet valve on the sample cylinder and purge the contents of the cylinder to atmosphere. Close the outlet valve and slowly open the cylinder valve attached to the manifold and fill the cylinder with source gas again. Close the manifold valve and purge (vent) the contents of the cylinder again to atmosphere. These steps are repeated based on the line pressure:

Psig Gas Pressure in Container	**Number of Purge Cycles**
15 – 30 psig	13
30 – 60 psig	8
60 – 90 psig	6
90 – 150 psig	5
150 – 500 psig	4
Greater than 500 psig	3

Once the appropriate amount of trap and purge cycles have been performed, the final volume is trapped in the cylinder and both valves of the sample cylinder securely closed at each end. Close the source pressure valve and bleed the pressure off the manifold by slowly opening the vent valve. After all of the pressure is off, disconnect the sample cylinder from the manifold. Make sure to obtain all operating data from the sample source, such as temperature, pressure, and flow rate, to include on your sample tag. Spray a small amount of your soap solution around the valve's stem and threads to check for leaks after sampling. If the valves show any sign of leaking, the sample should be taken again in a different cylinder. Wipe off any excess soap water and place a small amount of Teflon tape over the threads of both cylinder valves. Place end caps over valves and secure gently with wrench. Attach the sample tag, with all pertinent sample location information, on the cylinder.