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An American National Standard

Standard Practice for Obtaining LPG Samples Using a Floating Piston Cylinder[1]

This standard is issued under the fixed designation D 3700; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the equipment and procedures for obtaining a representative sample of specification liquefied petroleum gas (LPG), such as specified in Specification D 1835, GPA 2140, and comparable international standards. It may also be used for other natural gas liquid (NGL) products that are normally single phase (NGL mix, field butane, and so forth), defined in other industry specifications or contractual agreements.

1.2 This practice is not intended for non-specification products that contain significant quantities of undissolved gases (N_2, CO_2), free water or other separated phases, such as raw or unprocessed gas/liquids mixtures and related materials. The same equipment can be used for these purposes, but additional precautions are generally needed to obtain representative samples of multi-phase products (see Appendix X1).

1.3 This practice includes recommendations for the location of a sample point in a line or vessel. It is the responsibility of the user to ensure that the sampling point is located so as to obtain a representative sample.

1.4 The values stated in SI units are to be regarded as the standard. The values provided in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices, and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1265 Practice for Sampling Liquefied Petroleum (LP) Gases (Manual Method)[2]

D 1835 Specification for Liquefied Petroleum (LP) Gases[2]

2.2 *GPA Standards:*[3]

GPA 2174 Obtaining Liquid Hydrocarbon Samples for Analysis by Gas Chromatography

GPA 2140 Liquefied Petroleum Gas Specifications and Test Methods

3. Terminology

3.1 *floating piston cylinder (FPC)*—a high pressure sample container, with a free floating internal piston that effectively divides the container into two separate compartments.

3.2 *maximum fill density (reduced fill density)*—the volume of a container occupied by the sample, usually expressed as a percentage of the total capacity.

4. Summary of Practice

4.1 A liquid LPG sample is transferred under pressure from a sample point to a floating piston cylinder. The floating piston cylinder (FPC) is designed to collect liquid samples with no vaporization by displacing a piston against a pressurizing fluid (usually an inert gas). The piston serves as a physical barrier between the sample and the pressurizing fluid, at the sampling pressure. The position of the piston at the end of sampling indicates the percent fill of the sample cylinder.

4.2 It is the responsibility of the user of this practice to locate the sample point at a suitable location where the product being sampled is a representative, single phase, homogeneous liquid.

5. Significance and Use

5.1 This practice allows the collection of a representative sample of LPG that may contain trace volatile components such as methane, nitrogen, and ethane. Sampling by Practice D 1265 may result in a small, but predictable loss of these lighter components. Practice D 1265 is suitable for collecting samples for routine specification testing, as the small loss of light components is not significant under Specification D 1835 specification requirements. Practice D 3700 is recommended whenever highly accurate determination of light components is required. For example, composition determined on samples collected according to Practice D 3700 may be used to establish product value of NGL mixtures (see Appendix X1).

6. Interferences

6.1 An interference in a sampling procedure is anything which compromises the integrity of the sample.

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.H on Liquefied Petroleum Gases.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] Available from Gas Processors Assoc., 6526 E. 60th Street, Tulsa, OK 74145.

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6.2 Sample point location can give a non-representative sample due to solid or liquid contaminants, two phases, storage tank stratification, and so forth.

6.3 Reactivity of steel surfaces can remove or chemically alter trace reactive components such as H_2S, COS, and mercaptan.

6.4 A lubricant that is soluble in LPG can contaminate the sample.

6.5 Pre-charge gas can leak into the sample due to worn or damaged seals or poor surface finish (see 8.1).

6.5.1 Consult manufacturer's guidelines for suitable procedure to verify a leak-free cylinder, such as pressure testing each side of the cylinder. It is also possible to analyze the sample for inert gas, or the inert gas for hydrocarbon, to detect leakage in either direction.

6.6 Failure to flush sample lines and *dead volumes* can result in contaminants in samples.

6.7 Sampling from stratified tanks, *dead* zones in flowing systems, or inappropriate time periods in composite sampling systems will result in non-representative samples.

7. Apparatus

7.1 *Floating Piston Cylinder (FPC)*:

7.1.1 *Construction*, typically fabricated from corrosion resistant 316 stainless steel, in accordance with the pressure vessel certification requirements in the jurisdictions in which it is to be used, and through which it will be transported. Protective internal coatings or surface treatments are acceptable provided that they do not adversely affect the free movement of the piston, or effectiveness of the seals (see Fig. 1).

NOTE 1—At present, there is no international approval process for pressure cylinders. Cylinders require appropriate approval in each jurisdiction in which they are used or transported.

7.1.2 *Volume of Sample*, as required by the tests to be performed, typically 400 mL (that is, 80 % of 500 mL sample cylinder at 15°C).

7.1.3 *Piston Position Indicator*—The FPC shall be equipped with a piston position indicator such as a magnetic follower, piston rod, or equivalent which can be used to indicate the sample volume to comply with the maximum percent fill (maximum fill density) allowed for storage and transportation.

7.1.3.1 Floating piston cylinders that are not equipped with a piston position indicator shall not be used without a procedure to allow the operator to verify fill density immediately after sampling prior to transport. Consult the authority having jurisdiction for acceptable procedures.

7.1.4 The cylinder shall include a mechanism to mix the sample in the sample chamber in case of stratified mixtures or water haze that may settle after sampling. This mechanism may be a mechanical mixer/vortex plate on a movable rod, a freely moving rolling ball or slider, magnetically coupled stirrer, or similar device. It is the responsibility of the user to provide sufficient mixing with the mechanism used to allow a representative sample to be withdrawn.

7.2 Lubricants used to lubricate or seal the floating piston, O ring seals, and other components shall be inert to LPG.

NOTE 2—DuPont Krytox AC or AD have been found to be suitable.

7.3 A safety relief device is required to prevent overpressure in the event that a cylinder becomes fully liquid filled (hydraulically locked) from either overfilling or liquid thermal expansion from excessive temperature increase.

7.3.1 A rupture disk or a self-resetting pressure relief valve shall be fitted to the cylinder to prevent overpressure from hydraulic filling due to temperature increase.

NOTE 3—DuPont KEL-F, or similar materials have been found to be suitable. PTFE or other materials that are prone to *creep* under pressure, and metal-to-metal valve seals are not recommended.

7.3.2 Users may not alter valves or safety relief devices that are part of a cylinder permit or exemption. (The USA has an exemption system and Canada has a permitting procedure for non-ASME or DOT cylinders.)

7.4 *Sampling System*—It is not possible to provide a single procedure that will be applicable for all sampling situations. Different procedures and sampling equipment may be required for sampling pipes, storage tanks, rail cars, trucks, and smaller storage vessels in order to obtain a representative sample (see 4.2).

7.4.1 Sample equipment and procedures shall be designed and used to obtain representative samples of a product, and to maintain sample integrity for the tests being performed. A typical sampling system for LPG flowing in a pipe is shown in Fig. 2.

NOTE 4—While not required by this practice, the use of a sample probe in a flowing line is recommended. The sample probe should be located on the top or side of a line, extending into the center third of the flowing stream. Sample points should not be installed on the bottom of a line, unless provisions are made to flush any accumulated debris from the sample point immediately prior to sampling.

7.4.2 Transfer lines, valves, pressure gages and so forth in the transfer system shall be corrosion resistant and designed consistent with maximum anticipated pressure (typically stainless steel). Experience has shown that the transfer lines should have a minimum internal diameter of 3 mm nominal (⅛ in.) and be as short as practical to minimize line blockage or sample vaporization, or both. The use of filters, dryers, needle values and so forth are not recommended, unless provisions are made to prevent excessive flow restriction and pressure drop. A T junction with a purge valve at the sample connection point is recommended to allow purging of the dead volume at the sampler connection. Flexible hose or tubing with adequate pressure rating may be used.

NOTE 5—While not required by this practice, the use of non-reactive and non-absorbtive materials is recommended, especially when sampling to determine trace levels of reactive or polar materials such as H2S and water.

7.4.3 Other piping arrangements may be acceptable, but may require different flushing procedures prior to sample collection. Typical sample loops are shown in Figs. 3 and 4.

7.4.4 Closed loop side stream samplers designed to minimize volatile light end losses during sampling may be used. The sample system shall be connected to on-line analysers or composite samplers in a manner that ensures sample integrity is maintained for the tests being performed.

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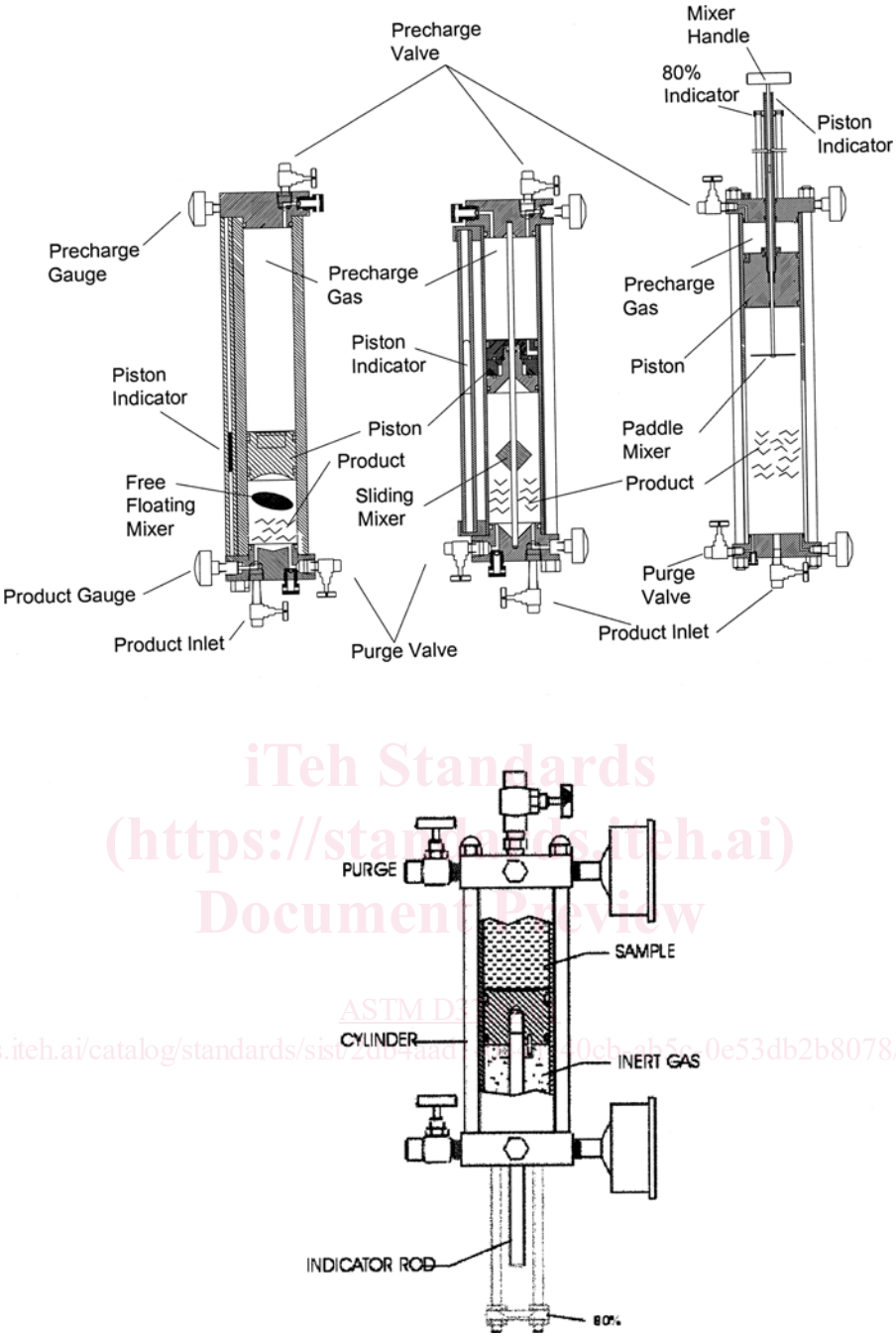

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FIG. 1 Typical Floating Piston Cylinders

7.4.5 Sample loops should preferably be installed around pumps, valves, or other sources of pressure drop to minimize atmospheric emissions from purging of sample lines.

7.4.6 Sampling pumps or other means of controlling pressures higher than the vapor pressure of the sample may be acceptable, and may be used to flush both the lines and/or the cylinder dead volume, if any, prior to sample collection. The cylinder may be partially filled and then emptied prior to collection of the sample as an alternative to venting hydrocarbon to flush lines.

7.5 *Composite Sampler*:

7.5.1 A composite sampler (also called a proportional sampler) is a device that is used to obtain a representative sample from a flowing product stream by accumulating small portions of product over a period of time. The sampling system consists of a sample probe, a means of collecting repetitive small portions, and a floating piston cylinder to accumulate the total sample. Figs. 5 and 6 show typical sample systems using a probe-mounted sample pump (Fig. 5), or a flow-through sample injection valve (Fig. 6).

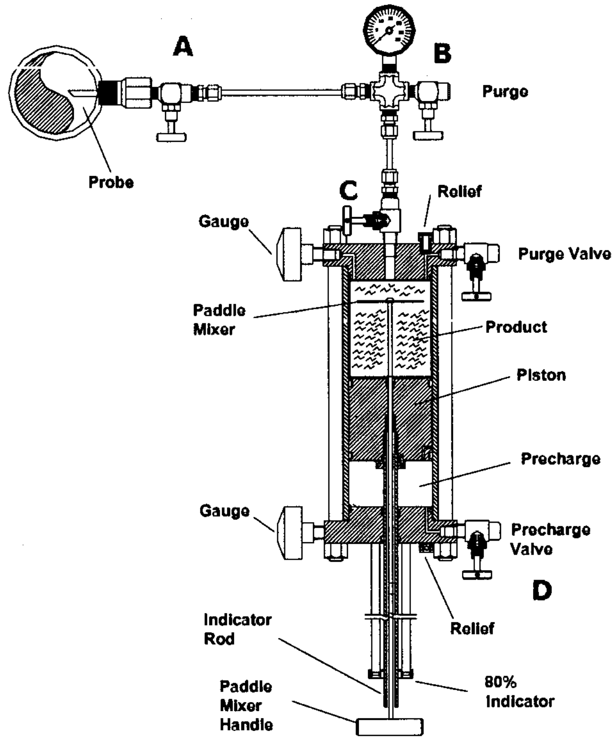

FIG. 2 Typical Sampling System

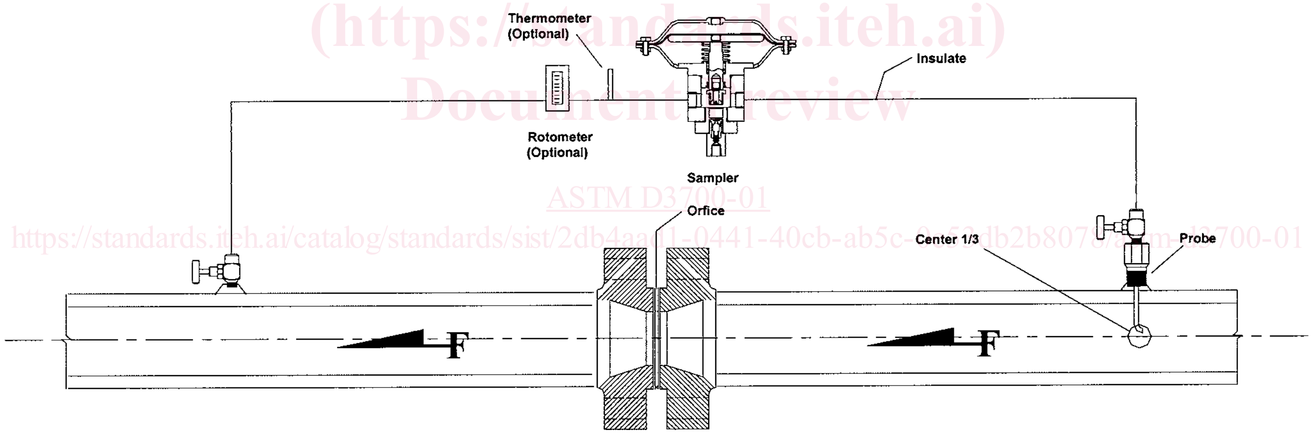

FIG. 3 Typical Sample Probe Installation on Orifice Fitting

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7.5.2 Thorough purging of sample lines, pumps, and connections to the sample cylinder is necessary to avoid contamination of the sample. A suitable purging arrangement at the point of sampling shall be provided. This purging arrangement may take the form of a T connection and purging valve, or an operating procedure that allows slight loosening of the connection to vent the dead volume at the sample connection (if allowed by the local jurisdiction). Composite sampler systems shall be designed to minimize dead end lines that could result in the sample not being representative of the LPG source.

7.5.3 Take precautions to avoid vaporization in sample loop lines when operating near the equilibrium vapor pressure of the LPG. In some instances, it may be necessary either to cool or insulate the sample line and sample container, or to control the pressure or temperature of sample containers.

7.5.4 Ensure that the pressure at the sample point is above the vapor pressure of the sample to avoid vaporization in the pipe or sampling lines when using a device such as an orifice plate or valve to create a pressure differential for sampling.

7.5.5 The floating piston cylinder shall be connected to the purged sample line of the composite sampler. Apply inert gas pressure to the cylinder to force the piston to the sample point end of the FPC. Maintain the inert gas pressure at a pressure that exceeds the equilibrium vapor pressure of the fluid sampled under the expected temperature conditions by about 350 to 1400 kPa (50 to 200 psi). At pressure differences less than about 1400 kPa (200 psi), there is an increasing chance of non-representative samples. Transient pressure fluctuation below the vapor pressure can result in non-representative samples.

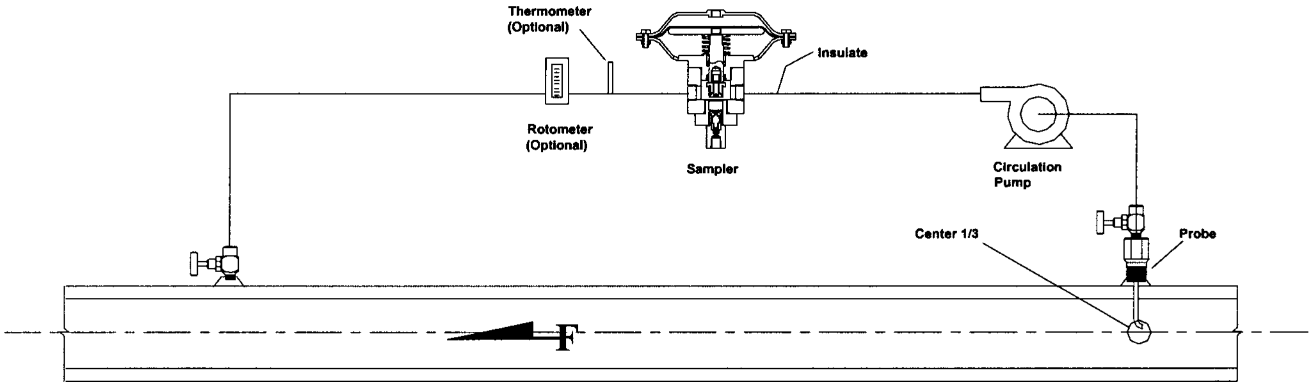

FIG. 4 Typical Sample Probe Installation for a Pump

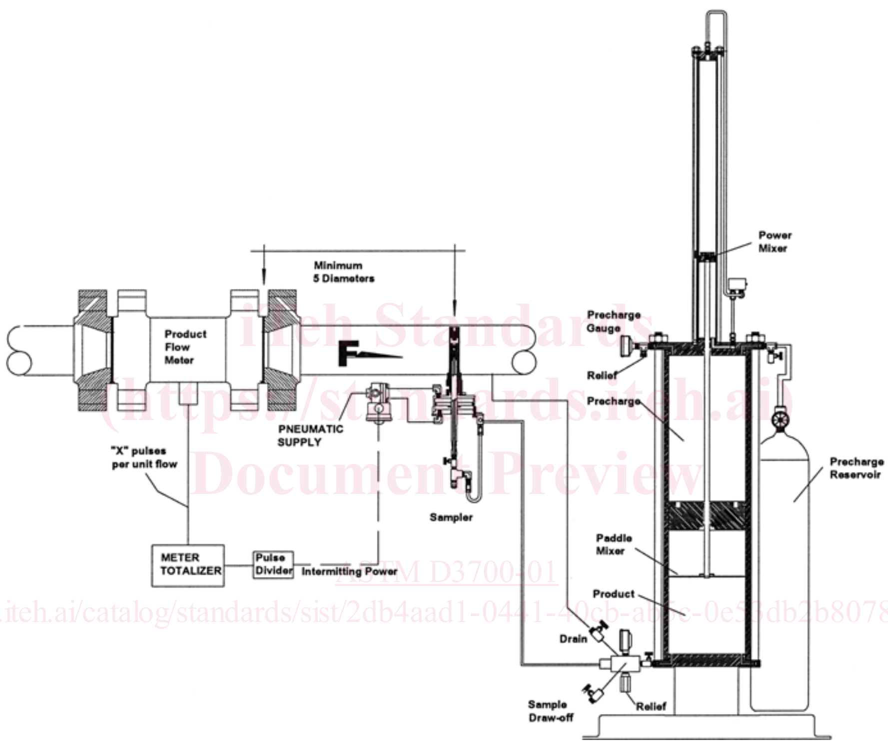

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FIG. 5 Typical Sampler Using an Injection Pump

7.5.6 Adjust the automatic sample injection valve to obtain incremental samples at a rate such that the floating piston cylinder will have adequate capacity to hold the sample during its period of sampling. Set the sampling rate to prevent overfilling of the sample container, considering the maximum anticipated flow rates and time periods. A liquid filled cylinder may not have a representative sample, since sampling may have stopped prior to the end of the sampling period, or preferential release of light ends (depending upon venting configuration).

7.5.7 Adjust the total volume taken over the sampling period in proportion to the flow rate of the product by adjusting each incremental sample volume taken by the sampling valve, or adjusting the frequency of incremental samples, or both.

7.5.8 The floating piston cylinder used in the above system may be removed from the composite sampling device after the desired sample has been collected.

7.5.9 Do not take outage or reduce pressure on the cylinder. Check valves for leaks, cap valves to protect threads, and prepare sample information tag and box for transport according to the Department of Transportation or other applicable requirements.

7.5.10 If it is not possible to disconnect the primary floating piston cylinder from the automatic system, mix the sample in the primary cylinder to homogenize it and transfer sample to a secondary floating piston cylinder. Proceed as in 9.2.1, treating the primary cylinder as a flowing source. (In this particular transfer situation, as sample is withdrawn, the master sample container will partially depressurize. Maintain the pre-charge pressure above the product vapor pressure at the existing master sampler temperature to prevent flashing.) (**Warning**—The cylinder must not be filled beyond 80 % of its capacity with sample. In the event of an inadvertent over fill, sample must be vented out to the required reduced fill density

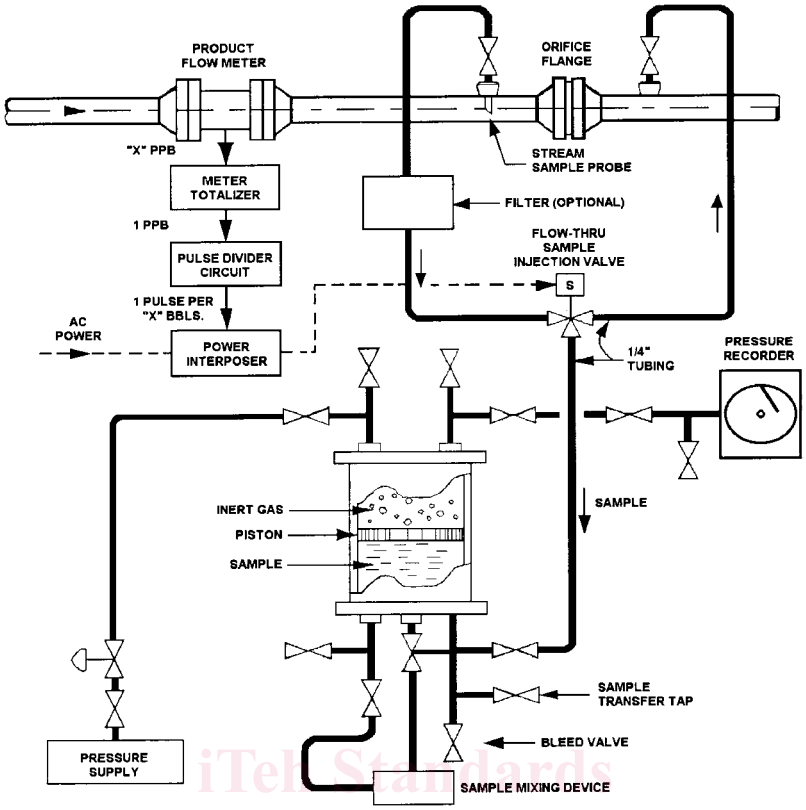

FIG. 6 Typical Automatic Proportional Sampler

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(typically 80 %) prior to transport. Where immediate venting is not possible; for example, inside hazardous locations or with toxic materials (especially H_2S), provisions shall be made to prevent temperature increase prior to venting in a safe location, transfer to a larger cylinder or immediate analysis or other disposition in accordance with the authority having jurisdiction. It is the responsibility of the user to establish safe procedures for use in permitted facilities that are not regulated by site permits or equivalent, separate from transportation regulations.)

7.5.11 Transfer and venting operations require careful control to maintain the cylinder pressure well above the vapor pressure of the well mixed sample to prevent changes in sample composition from *flashing* of the contents.

7.5.12 *Sample Filters*—The sample filter is an optional device used to protect the sampling valve from scoring due to the presence of foreign contaminants such as metal shavings, dirt, and so forth. The filter should be of a small total volume, of an inline-type design, and contain a replaceable/disposable element. (**Warning**—Be aware that using filters or strainers can entrap water and other components which could result in an inaccurate analysis. Avoid use of filters or strainers if possible.)

7.5.12.1 *Water Accumulation*—Filters or other devices shall be oriented so that any free water passes with the sample and is not accumulated in the filter housings and so forth, leading to false low collection if water is present. This is critical for butane, where accumulation of water in the sampling system may cause false pass of the butane dryness criteria (no free water by visual inspection of the sample). Since specification propane must be subsaturated with water to pass the dryness criteria, any free water in propane will cause failure of the dryness criteria.

8. Reagents and Materials

8.1 The pre-charge gas should be an inert gas such as helium, nitrogen, or argon.

8.1.1 The preferred pre-charge gas is one that is not normally present in the sample (helium, nitrogen) so that it can readily be detected in the event of leakage, or that will not be detected in the analysis to be done. Helium is used as a carrier gas in GC analysis. Nitrogen or helium is not detected by flame ionization detectors, but will be detected by other types of GC detectors.

8.1.2 The use of natural gas (methane) or ethane as a pre-charge gas is not recommended because any leakage would increase the methane/ethane content and ratio, and increase the vapor pressure of the sample. This will give erroneous results that are not easily recognizable, due to the normal trace presence of these materials in LPG.

9. Procedure

9.1 *Preparation of Sample Cylinder*:

9.1.1 Thoroughly clean cylinders prior to initial use or after *change of service* or repair, with an appropriate cleaning agent, following the manufacturer's recommendations. Remove any traces of cleaning agent by evacuation, gas purge, or solvent wash, as appropriate. The use of steam is not recommended for the cleaning of floating piston cylinders.