CALIBRATION STANDARD GASES

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Introduction

In today's natural gas industry, it is imperative that the standards used to calibrate are made to the utmost quality. Calibration standards are mixtures of known concentrations of components used to confirm or determine concentrations in samples. Calibration standards are needed for quality assurance/quality control, measurement and balance, quantitative sample analysis, and custody transfer. Preparation, blending, and final analysis are all crucial factors which will determine the integrity of the calibration standard. Several guidelines, such as ISO, GPA, and API must be followed in order to produce a calibration standard that is accurate and consistent.

Preparation

All manufacturers should have a written quality assurance protocol for cylinder preparation and blending methods. DCG evacuates all cylinders prior to filling using a DCG proprietary protocol developed and validated that removes trace residual components. Due to the many different calibration standards manufactured, the specific protocol for evacuation can be from a basic vacuum purge to a complex, internal cleaning.

Figure 1. Vacuum station

Blending

According to the American Petroleum Institute (API), calibration standards must be, at a minimum, gravimetrically prepared and traceable by weight to the National Institute of Standards and Technology (NIST), or equivalent party. NIST Traceability validates the link between the calibration weights. In addition, any raw materials used in the blending process must be analyzed for impurities which must be considered in the making and analysis of the standard.

All balances used in the production of calibration standards must be calibrated and monitored daily and linearities verified weekly. In addition, bi-yearly evaluation by an outside metrological laboratory meeting ISO Guide 25 and ANSI/NCSL Z540-1 is required. Weights should be kept in protective cases and handled in such a manner to avoid soiling from dirt and oils which may provide a false calibration.

Figure 2. Balances and masses used to gravimetrically prepare calibration standards

Certification

After the blend is mixed thoroughly it is time to certify that the blend has met all the method specifications or requirements. Our mixtures are analyzed and compared to our seven-point calibration curve. These analyzed values are compared to the gravimetric values and are used to confirm the gravimetric values. These blends are also checked for repeatability and reproducibility in accordance with any requirements.

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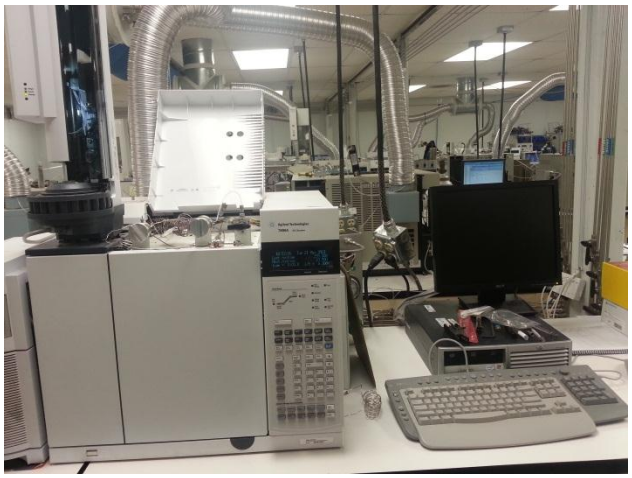

Figure 3. Gas Chromatograph set up for the certification process

Maintenance

To maintain the integrity of the calibration standard, proper maintenance is key. Calibration standards represent a significant investment of time and money. Exposing calibration standards to temperatures below their hydrocarbon dew point may cause stratification, causing the heavier components to settle to the bottom of the cylinder, while the lighter components collect near the top. Opening the cylinder valve in this condition will release the lighter components, altering the composition of the calibration standard. When this occurs, the quality of the standard is compromised and each component's actual concentration is changed. This results in erroneous analytical data and a ruined calibration standard. API 14.1 defines how calibration standard maintenance via heating the standard is done. It is recommended that the calibration standard be heated for at least 4 hours after the cylinder has reached a temperature of 30°F (17°C) above the expected hydrocarbon dew point of the calibration standard. Used properly, heat blankets will prevent the above from happening by heating the cylinder to the desired temperature.

The sample lines from the calibration standard to the gas chromatograph and any regulators being used in the sampling process must also be maintained at a temperature of at least 30°F (17°C) above the hydrocarbon dew point. This can be accomplished with heat trace tubing; heat trace tubing assists in maintaining the integrity of the standard during sampling.

When drawing a sample, if the ambient temperature is below the hydrocarbon dew point, condensation of the standard within the sampling system lines may occur. Electrical heat trace tubing is used to heat the sampling system lines to avoid this. Heating the regulator offsets the reduction in temperature associated with pressure regulation.

Figure 4. Heat blanket

Conclusion

In an industry where business is based on the quality of the final product, a calibration standard that is prepared to the highest caliber is critical. This imparts confidence to companies that the calibration standards they have purchased are accurate and precise and will fulfill their contractual obligations. An incorrect or lesser grade standard will result in the imbalance of the purchasing plant or pipeline. However, it does not end there. To preserve the reliability of the calibration standard and guarantee the maximum shelf life, proper care and maintenance is a must.