

LOW CONCENTRATION CALIBRATION FOR HIGH VAPOR PRESSURE GASES



Creating trace calibration gas standards for high vapor pressure atmospheric gases such as O₂, NO, CO, N₂, CH₄, and others has driven the requirement for using permeation tubes to achieve repeatable NIST Traceable gas sources. Since 1966, permeation tubes have been a proven method of calibration for trace concentration gases after O'Keefe, and Ortman published early findings relating permeation tubes as a primary standard used for trace gas analysis (7). Since then, environmental, air monitoring, petrochemical, and other applications have improved detection capabilities that require lower concentration gas standards beyond what is typically feasible in a gas cylinder mixture. Gas cylinder mixtures of some air pollutants are reactive or unstable and storing a static cylinder mixture for extended calibrations results in inaccuracies (2). Permeation tubes are an effective method for creating NIST Traceable gas standards (1). They provide a stable means of dynamically generating accurate gas standards. The advantage is that the permeation tube method can be used with a wide range of analytes including condensed gases, liquids, subliming solids, and permanent gases (5, 8). Currently, over 550 analytes are available in some type of permeation tube for delivering trace gas concentrations (3). Most commercially available permeation tubes are filled with a pure liquid analyte (or gases condensed to a liquid). Those analytes such as permanent gases that cannot be liquefied pose a unique challenge because they must be used in pure gas form and require the right instrumentation and controlled parameters for use in a permeation tube.

*"Permeation tubes are an effective method
for creating NIST Traceable gas standards."*

Calibration Challenges Solved with Permeation Tube Technology:

KIN-TEK Analytical, Inc. specializes in providing solutions to calibrate highly sensitive gas analyzers, sensors, or detectors using permeation tubes and permeation systems (gas standard generators). The following application demonstrates the challenge of dynamically creating trace calibration gas standards (ppm and lower) with high vapor pressure and permanent gases. A solution is posed using a permeation tube and instrument to "feed" gas into the tube at controlled parameters to allow equilibrium and permeation to occur.

Challenge:

The challenge is that pure gases considered permanent gases or air pollutants that cannot be liquefied, such as O_2 , NO, CO, N_2 , CH_4 , NO, etc. must be used in the gas form for calibration and are unstable in static mixtures at trace concentrations. Using a permeation tube for emission at a very low flow rate for further dilution to ppm and lower concentrations requires precision flow, pressure, and temperature control devices. Dynamically generating the gas mixture as it is needed eliminates issues created from storing the mixture.

Solution:

Trace Source™ 57H Gas Fed permeation tubes used in a FlexStream™ Base and PM/GF Module provide the controls needed for creating ppm and lower concentrations of high-pressure and permanent gases such as O_2 , NO, CO, N_2 , CH_4 , and other gases that cannot be liquefied. The mechanism and controls needed to deliver accurate gas standards at trace concentrations is a combination of the tube construction and the equipment controls required to hold the tube at equilibrium conditions.

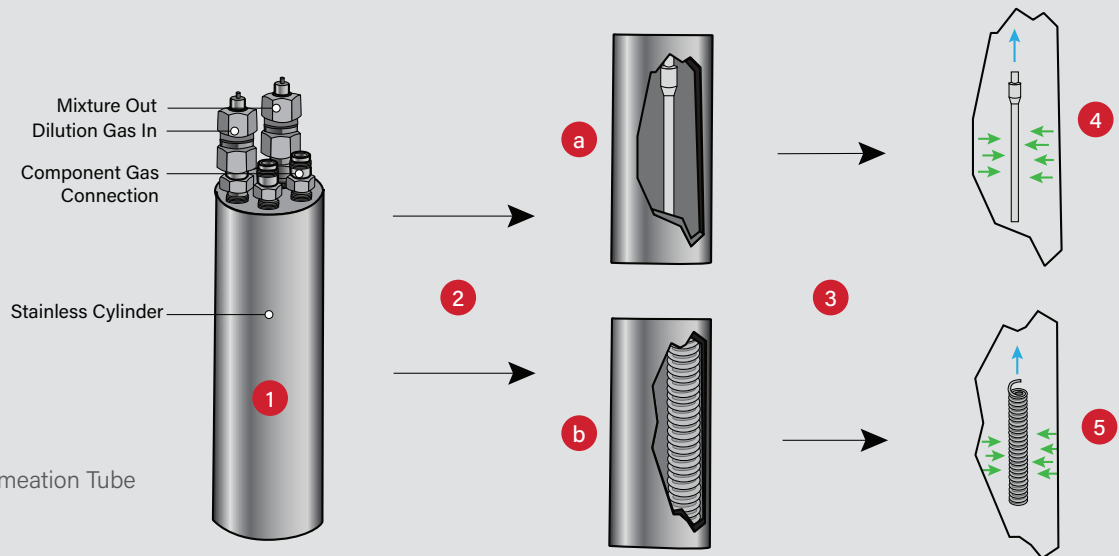


Figure 1 Trace Source™ Permeation Tube
How it Works

- 1 Trace Source™ 57H or 57S Gas Fed Permeation Tubes are typically made with a stainless-steel body that connects inside a KIN-TEK instrument via adapter fittings for feeding pure component gas directly into the tube.
- 2 An internal membrane made of material such as TFE (tetrafluoroethylene) or FEP (Fluorinated ethylene propylene) Teflon provides a physical barrier to the pure component gas.
 - a. A short thick-walled membrane is used for concentrations of ~10 ppm and lower in a 57S Gas Fed tube.
 - b. In a 57H permeation tube a smaller diameter, longer membrane (up to 2000 cm) is used for higher ppm concentrations up to ~1000 ppm, (depending on the analyte component gas).
- 3 In the Gas Fed Permeation Tubes, gas is fed into the tube by instrument controls and surrounds the inner membrane. Permeability takes place through the membrane based on the product of the diffusivity and the solubility of the pure component gas in the membrane and is a function of temperature (5).
- 4 The factory certified tube emission rate (given in nl/min) constitutes a very low controlled flow of the gas across the membrane and depends on the difference in analyte partial pressure between the inner and outer membrane walls of the permeation tube at a precisely controlled temperature (5, 6).
- 5 A small flow of dilution gas circulating through the internal membrane picks up the permeating gas and carries it through the system for further dilution and delivery to the analyzer (2).

How Trace Source™ Gas Fed Permeation Tubes Work with the FlexStream™ System

The FlexStream™ System, specifically the FlexStream™ PM/GF (Gas Feed) Module offers a permeation oven with enhanced capabilities for holding the permeation tube at a stable, precise temperature and pressure. The unique feature of the FlexStream™ PM/GF that differs from a typical permeation oven is a pressure control subsystem and a component gas flow path for safely feeding pure component gas from a cylinder directly into the permeation tube (4). See **Figure 2**.

An internal pressure controller maintains the pure component gas in the tube at a set pressure, slightly above ambient. An isolation valve and purge control valve allow the user to fill, purge, and refill the permeation tube as often as needed, always having a fresh gas standard for calibration. The certified emission rate of the tube determines frequency of filling and purging the pure gas from the tube. Purge controls are used when the emission rate of the tube is high, but not so often if the emission rate of the tube is low. Once filled with purge gas and stabilized, the permeation tube operates as any other permeation tube. The FlexStream™ system controls the operating temperature, the partial pressure drop across the permeation tube membrane, and the dilution gas flow through to the analyzer (2).

Operation of the FlexStream™ instrument is easy for users that want to avoid the hassle of calculating the settings needed to achieve a multi-point calibration curve. Multiple modules can be placed in a series with the Base Module as the controlling module. **Figure 3** shows a FlexStream™ Base Module and FlexStream™ PM/GF Module placed in a three module expansion cabinet. In this scenario, up to 8 disposable tubes can be installed in the FlexStream™ Base Module and one Trace Source™ Gas Fed tube installed in the FlexStream™ PM/GF module for creating complex mixtures of gases at various concentrations. Toggling one or both ovens on extends the system capabilities for more defined selections.

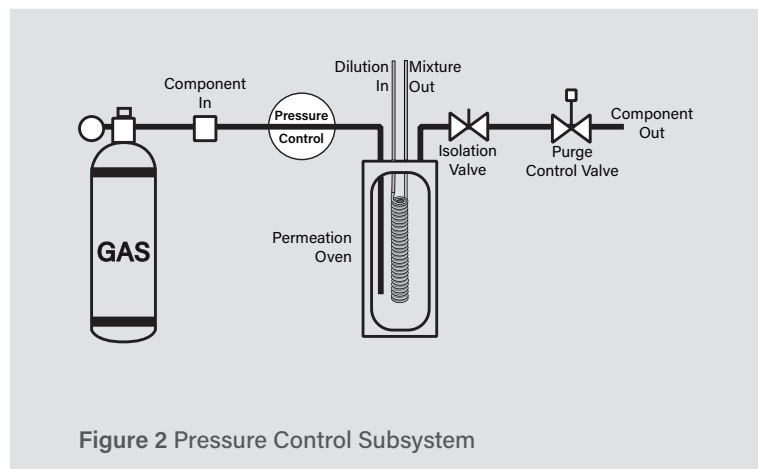


Figure 2 Pressure Control Subsystem

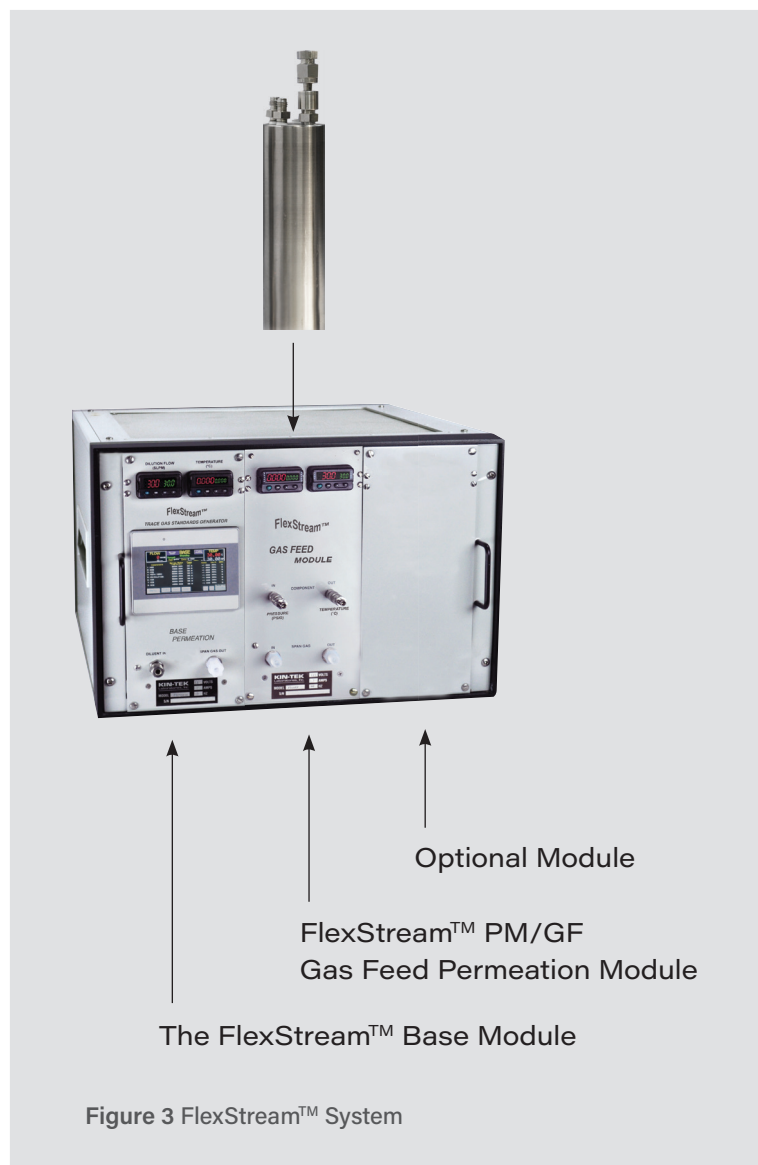


Figure 3 FlexStream™ System

Details of the permeation tube are entered into the instrument "Tube Setup" touchscreen menu, such as the certified emission rate at the equilibrium temperature and pressure. The system uses the information to calculate concentrations based on the desired span gas method. See **Figure 4** for Tube SetUp. For example, the Span-by-Flow method calculates the output concentration based on the user flow settings and starts creating calibration gas. The Span-by-Concentration method calculates and sets the flows needed to generate the desired concentration. **Figure 5** shows an example of the FlexStream™ PM/GF touchscreen readout of Span Gas at 0.999 PPM being generated using the target gas COMP 1 held at a pressure of 16.07 psia and Temperature of 30.01 °C. The tube emission rate is 1000 ng/min at 30.00 °C as provided in the Tube Setup Screen.

One Trace Source™ 57H Gas Fed Tube installed into a FlexStream™ System replaces several gas cylinders and avoids calibration issues related to static blends of high vapor pressure gases such as O₂, NO, CO, N₂, CH₄. Trace Source™ refillable permeation tubes provide years of service and can be returned annually to the factory for recertification for NIST traceability. One tube literally replaces the need for years of cylinder gas purchases. Additionally, FlexLink™ Software included with the FlexStream™ System allows users flexibility by providing computer access to all instrument controls and logs data for monitoring concentrations, flow, temperature, etc. Hence concentration changes are done with the touch of a button and a multi-point calibration curve can be made in a matter of minutes. The use of this system prevents the need for static cylinders.

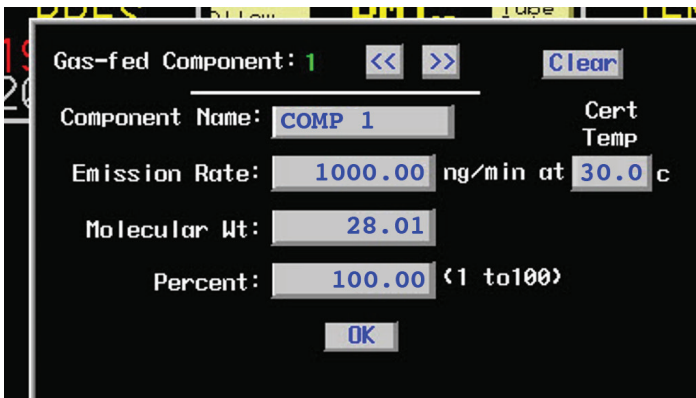


Figure 4 Tube Setup Screen

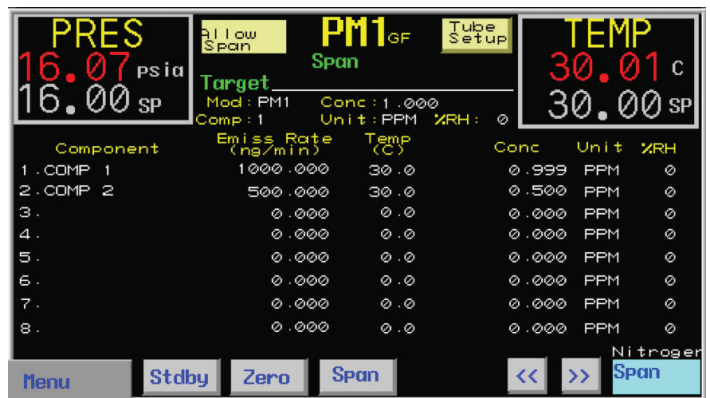


Figure 5 FlexStream™ PM/GF Screen

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Solve Your Calibration Challenges with KIN-TEK Analytical Inc. Products

The Trace Source™ Permeation Tube technology is employed in KIN-TEK's Gas Standard Generators to provide accurate, NIST traceable calibration standards. KIN-TEK's products include a range of gas standard generators and permeation devices to fit almost any application that relies on the delivery of an accurate trace gas concentration. Individual gas generator modules can operate as stand-alone calibrators or be combined into a Gas Standard Generator System configured to solve the most complex applications. The System utilizes the FlexLink™ software that can log and export data for analysis and reference.

Contact a customer service representative now and discuss your specific application.

KIN-TEK 
The Calibration Specialists

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