Sample Prep Perspectives

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This month's "Sample Prep Perspectives" looks at methods for preparing calibration standards for volatile organic compound analysis. Guest author Jim McKinley and column editor Ron Majors compare traceability for static and dynamic methods such as direct mechanical dilution, diffusion and permeation tube extraction, exponential dilution, and electrochemical extraction.

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The Preparation of Calibration Standards for Volatile Organic Compounds — A Question of Traceability

The analysis of trace volatile organic compounds (VOCs) has been of considerable interest for decades, particularly for monitoring air emissions, ambient air, and indoor air contamination in the workplace. The combination of new gas chromatography (GC) column technology with improvements in GC instrumentation, especially in detector sensitivity and selectivity improvements, has led to routine measurement and detection of sub-parts-per-billion concentrations. Researchers have directed much effort toward decreasing detection limits and achieving repeatability. As measurement technology improves, users demand lower levels of detection. In the chemical and petrochemical industries, regulatory and competitive demands are forcing the ever-increasing use of on-line analysis for process and product quality control. As specifications become tighter, the concentration levels move lower. On-line measurements of parts-per-million and parts-per-billion concentrations have become commonplace. Modern quality assurance and quality control systems ensure that a certain product or process meets a certain standard or level of quality. Certainly method validation has become a standard practice driven by quality initiatives such as ISO 9000.

All quality systems require traceability of accuracy to accepted national standards and the routine demonstration of measurement validity (1). Verification of a chemical standard against a second source is a sound technical practice, as well as a legal requirement in many cases. It provides confirmation of compound identity, purity, and stability. The second source may be a certified reference standard or a substance that has been compared with a certified reference standard material. The comparison or verification of standards should be performed under the exact conditions used for the calibration of the instrument. Ideally, calibration should be performed in a similar matrix or environment that will be used for a sample.

This month's "Sample Preparation Perspectives" will examine the issue of generating standard mixtures of VOCs for GC analysis, particularly for the purposes of achieving traceability. We will compare various approaches and cite their strengths and weaknesses.

Achieving Traceability

The term *traceability* has different meanings in different contexts. In calibration, traceability refers to the ability to compare data with an accepted standard. The International Vocabulary of Basic and General Terms in Metrology (VIM) defines traceability as "The property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties" (2).

In the United States, national standards are set and maintained by the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland. NIST maintains vertical traceability to the International System of Units (SI) and horizontal traceability with other national laboratories around the world. The SI system has two base units that are of relevance in chemical analysis: the unit of mass (gram, abbreviated g) and the amount of chemical substance (mole, abbreviated mol). For commercial use, NIST offers standard reference materials that can be purchased for use as resident standards.

Although defining traceability is simple, obtaining traceability of an analytical measurement, especially at trace concentrations, is not as straightforward. Under the VIM definition, the clearest path to traceability is calibration to an NIST standard reference material or to a standard that is traceable to a standard reference material. The problem is the discrepancy between the range of

standards available and the range needed for analysis. Currently, more than 400 VOCs of environmental and industrial interest are measured as vapors at trace concentrations (parts per million, billion, or trillion). Yet, we have trace-level vapor standards for only 30 compounds or so. Most of these compounds are halocarbons and are available at single concentrations only. Establishing calibration accuracy for hundreds of VOCs is a significant problem, because NIST guidelines require a candidate concentration for comparison to be within ±5% of the standard reference material concentration. Very little work has

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been performed to develop the needed standards that are traceable to NIST standard reference materials.

Thus, for most compounds, the only route to traceability is using fundamental standards directly. If the concentration of a mixture can be related through physical variables such as mass, length, or time for which standards exist, then analysts can use the traceability of the measured values to those variables to obtain the traceability of the resulting concentration. As we discuss the methods of calibration-standard preparation, we will relate them to their potential for traceability and then discuss and compare them later.

Sources for Trace Concentration Gas Standards

Analysts can use many methods to prepare standard mixtures of gaseous samples; some are simple and others are very complex. They can be roughly classified as static methods and dynamic or blended methods.

Static methods: Static samples are prepared by taking a known weight or volume of volatile organic gas or liquid and placing it in a container of known volume. These samples are prepared easily, everybody can understand how the procedure works, and minimal equipment is required beyond a volumetric device or a balance and a known-volume container. These devices are

portable and can be taken to the field or to the factory floor. Essentially, the static method is goof proof — the accuracy is independent of user competence, and this method can be used by very inexperienced personnel.

Static methods are not without pitfalls, however. The sources of error are not well defined. As the concentration of an individual component decreases, the stability of the standard mixture often decreases. For example, polar or reactive components can interact or adsorb on container walls. High molecular weight or high boiling point compounds can condense and also be lost within the container. The preparation of reactive mixtures is impossible because of prolonged contact and chemical reaction. Only dry mixtures are possible because humidity should be avoided. Static mixtures can be prepared at single concentrations only. For multipoint calibrations, analysts must use multiple containers, each of which has its own individual concentration; otherwise the base mixture requires an accurate dilution, which adds a level of complexity to the standard preparation.

When working with a trace concentration static mixture, the traceability of the variables measured during the preparation of the mixture does not imply traceability of the delivered concentration. This type of traceable preparation technique generally is not recognized because of possible interactions or changes that may occur within the cylinder during storage. Thus, for the preparation of static standards, traceability of the delivered concentration requires comparison to a standard reference material or other traceable reference standard. The distinction between traceability of concentration and traceability of preparation is poorly understood in the analytical community, and vendors often gloss over this distinction when responding to inquiries about traceability.

Dynamic methods: A wide variety of dynamic methods is available for the preparation of standards. Each of the methods has varying degrees of traceability. The sidebar "Dynamic Blending Methods" lists these methods. We will discuss the principles of the main techniques.

Direct mechanical dilution: Dynamic mechanical dilution is associated with two operational strategies: dilution of pure compounds and dilution of mixtures. In these strategies a steady stream of analyte vapor is directed into a flowing stream of inert gas. Figure 1 is a flow schematic of a mechanical dilution system. The concentration of each

analyte in the mixture is the ratio of the flow of that analyte to the total mixture flow, as shown in equation 1:

$$C_1 = \frac{f_1}{F_1 + f_1 + f_2 + f_3 + \ldots} \quad [1]$$

where C_1 is the concentration of Analyte 1; F_1 is the matrix gas flow; and f_1, f_2, and f_3 are the flow rates of Analytes 1, 2, and 3. If each of the mass flow-rate measurements is traceable, then the concentration of each analyte is traceable. Usually, electronic thermal mass flowmeters are used to measure the gas flows. These flowmeters can be calibrated to within 1% of reading accuracy levels and can hold their calibrations for long periods of time (3,4).

Mechanical dilution calibrant preparation avoids the storage stability problems associated with static mixtures. The error sources are reasonably well defined. Analysts can prepare both reactive and humid standard VOC mixtures. The preparation of multipoint calibration standards requires only a dilution gas or analyte stream flow rate change. One key advantage of direct blending is that it can be used throughout a very wide dynamic range and can generate higher concentrations than some other methods. Commercial products are available to prepare these dynamically created calibrants, and the technology is well developed. After startup, equilibration is rapid and calibration standards can be used right away.

Achieving the required accuracy requires sophisticated flow measurement of input gas streams with direct mechanical dilution. First, the flowmeters must be calibrated with the actual gases to be metered. Second, the calibration must be performed against dilution gas and analyte vapor primary standards. It is difficult to determine traceability because of the limited range of available flow standards and calibration gases. Because flowmeters are calibrated with a surrogate gas for which a primary standard exists, users must apply a correction factor to the primary calibration to obtain calibration for the actual gas used. Because of these uncertainties, most mass flowmeter measurements are closer to ±5 % or more of full-scale accuracy rather than the theoretical ±1% (3).

Generating accurate trace concentrations requires multiple stages of on-line dilution, which presents some practical mechanical problems. It is very difficult to prepare mixtures of liquid analytes because of the problem of creating a steady flow of analyte

vapor from a liquid source. Furthermore, the production of calibration mixtures that contain large numbers of analytes is impractical unless the approach is dilution of a multicomponent static mixture. The direct mechanical blending technique is not goof proof — user technique can affect mixture accuracy.

Diffusion tube: The diffusion tube method makes uses of the phenomenon that a source of vapor at constant temperature and pressure contained at one end of a tube will flow by diffusion down the tube. If the vapor pressure at the exit end of the tube is much lower than at the feed end, a steady and predictable flow results. Figure 2 shows a schematic of a diffusion tube. A reservoir at one end of a precision-bore capillary tube holds a small sample of liquid or solid analyte. At constant temperature, the vapor pressure of the analyte forms the constant pressure vapor source. A flowing stream of matrix gas passing over the other end of the capillary mixes with the diffusive vapor flow from the analyte to form the calibration gas mixture. Usually, the entire device is immersed in the matrix gas flow, as Figure 3 depicts.

The flow rate from a diffusion tube is given by equation 2:

$$E = 2.216 \times 10^6 \frac{DMPA}{TL} \log\left(\frac{P}{P-p}\right) \quad [2]$$

where E is the emission rate in nanograms per minute, D is the diffusion coefficient at temperature T and pressure P in centimeters squared per second, M is the molecular weight, P is the total pressure in torr, A is the cross-sectional area of the diffusion path in centimeters squared, T is the temperature in kelvins, L is the length of diffusion path in centimeters, and p is the partial pressure of the analyte

vapor at T in torr.

In equation 2, the diffusion coefficient D also is a function of temperature and pressure as shown in equation 3:

$$D = D_0(T/T_0)^m(P_0/P) \quad [3]$$

where D_0 is the diffusion coefficient at standard temperature and pressure, T_0 is 273.1 K, P_0 is 760 torr, and m is a constant, usually 2 but can be 1.75.

Analysts can use equations 2 and 3 to estimate the output expected from a diffusion tube, but the actual measurement is required for accurately known mixtures. The emission rate is determined gravimetrically by holding the diffusion tube at constant operating conditions and periodically weighing it to determine the rate of analyte weight loss.

Equation 4 yields the concentration of a mixture prepared from a diffusion tube:

$$C_i = \frac{E_i K}{F} \quad [4]$$

where C_i is the concentration of analyte i in parts-per-million, E_i is the emission rate of analyte i in nanograms per minute, F is the dilution flow in milliliters per minute, and K is the conversion factor to convert nanograms per minute to nanoliters per minute at standard temperature and pressure.

The flow emitted by the diffusion tube depends on temperature and pressure. Typically a 0.2 °C change in one or the other parameter causes an approximately 1% change in flow from the diffusion tube. To achieve traceability, users must precisely control several experimental parameters with traceable measurements; namely temperature, weight loss from the diffusion tube, dilution gas flow rate, and total pressure. It goes without saying that analyte purity is of utmost importance.
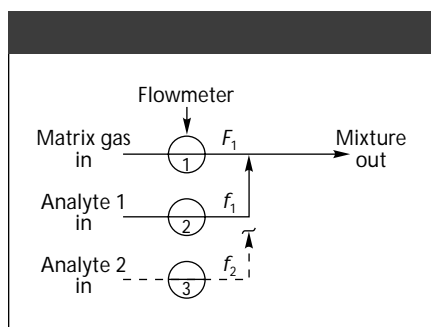
With diffusion tubes, it also is important to ensure that equilibration has occurred. Abnormal diffusion rates can result, however, if the internal surface of the capillary tube is contaminated with analyte liquid or solid. Once stabilized, diffusion equilibration should be concurrent with temperature equilibration.

The advantages of diffusion tubes are their low cost and ease of use for creating low concentration, one-shot mixtures for screening or survey applications. Equilibration is rapid. This approach is good for low-vapor-pressure semivolatile liquids as well as heat-sensitive compounds. Diffusion tubes exhibit higher emission rates than permeation tubes, which we will discuss below. By using a recording balance or occasionally weighing the diffusion tube, analysts can monitor the emission rate during use, thereby increasing mixing accuracy during delivery.

Diffusion tubes are unsuitable for gases or pressurized mixtures. The interfacing requires careful attention to achieve constant pressures and temperatures, and error sources are not well defined. The diffusion tube method requires a high level of operator skill to achieve successful results.

Permeation tube: In the permeation tube method, an analyte is held within a container on one side of a permeable membrane, which usually is polytetrafluoroethylene or fluorinated ethylene–propylene copolymer although other materials could be used. This method is similar to the diffusion tube method in that both techniques require analyte flow from a self-contained reservoir controlled by molecular dynamics. With permeation tubes, the flow is controlled by the permeability of a membrane to the analyte rather than diffusion across an open channel in a capillary tube, as is the case for diffusion tubes.
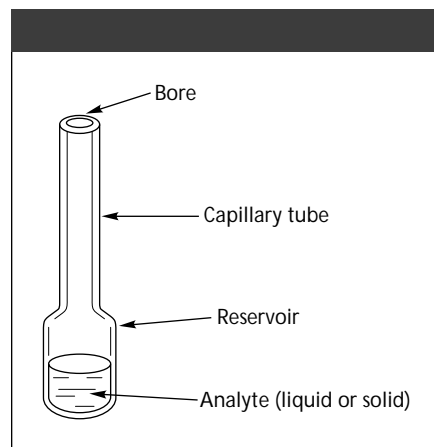

Figure 1: Flow diagram for a direct gas blending system for preparation of standard calibration mixtures.

Figure 2: Schematic of a diffusion tube.

Figure 4 is a schematic of a typical permeation tube. A small quantity of gas, liquid, or solid is sealed inside a short length of plastic tubing. The tubing wall serves as a permeable membrane separating the pure analyte from the matrix gas flow. Any analyte permeating through the tubing wall creates a small, very stable flow of analyte vapor. Mixtures are created by immersing the tube in a flow of matrix gas, as shown for a diffusion tube in Figure 3.

The permeability of a membrane to a compound is defined as the product of the diffusivity of the compound through the membrane and the solubility of the compound in the membrane as stated in equation 5:

$$K = DS \qquad [5]$$

where K is the analyte permeability, D is the analyte diffusivity, and S is the analyte solubility.

The flow rate of analyte vapor from a permeation tube is given in equation 6:

$$f = K_T \left(\frac{A \, \Delta P}{t} \right) \qquad [6]$$

where f is the flow rate of the analyte emitted by the tube, K_T is the permeability of the membrane to the analyte at temperature T, A is the membrane area in which permeation occurs, ΔP is the partial pressure across the membrane for the analyte vapor, and t is the membrane thickness.

The permeability K varies exponentially with absolute temperature as shown in equation 7:

$$K(T) = \beta e^{-\alpha/T} \qquad [7]$$

where α and β are constants and T is the absolute temperature in kelvins.

The flow of emitted analyte usually is determined gravimetrically by the same method used for diffusion tubes. In fact, equation 4 also pertains to permeation tubes. The requirements for maintaining traceability are similar to those for diffusion tubes. The temperature of the permeation tube, the flow rate of the matrix or dilution gas, and the measurement of the emission rate must be traceable. One major difference compared with diffusion tubes is that the total pressure is not a factor of concern, but analyte purity is central to mixture accuracy.

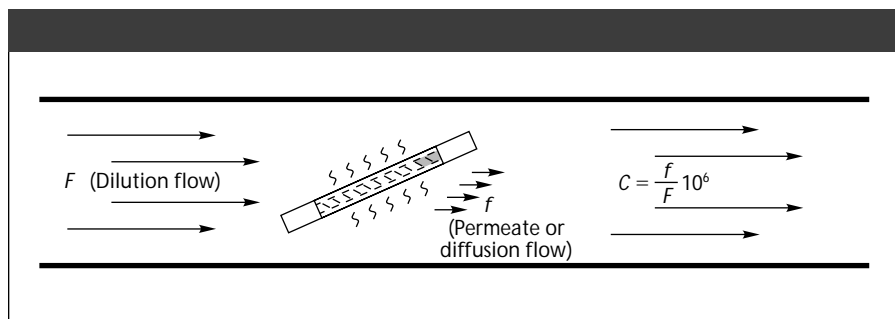The key advantage of permeation tubes is the range of compounds for which the

Figure 3: Flow schematic for diffusion and permeation tube systems.

method can be used, including condensed gases, permanent gases, liquids, subliming solids, and many semivolatile organic compounds. The two major constraints for analytes used in permeation tubes are that they must be available and stable in pure form and that they must have long-term stability at a temperature that will create at least 1 mm of mercury vapor pressure. High vapor pressure analytes can be handled in special permeation tubes designed to use compound vapor as the source of analyte. Permeation tubes avoid some of the storage stability problems of static mixtures.

Permeation tubes also offer high accuracy for a wide range of concentrations. Concentrations from more than 5000 ppm to less than 50 ppb can be determined in a single dilution step. Multipoint calibration requires only a flow-rate change. Combining permeation tubes in parallel can generate multicomponent mixtures. Concentrations are easily traceable through physical standards for most analyte compounds. The emission rate can be monitored by periodic weighings throughout the life of the tube ensuring mixture accuracy at delivery. The error sources in permeation tube systems are reasonably well defined.

Because constant analyte delivery depends upon equilibration, the primary limitation of the permeation tube method is the speed of equilibration. Prestabilized permeation tubes typically require 2–24 h to equilibrate following a cold startup. Achieving accuracy requires sophisticated flow and temperature measurement. The permeation tube concept is not widely understood, and its successful use requires operator education.

Permeation tube systems produce a continuous flow of calibration mixture rather than a one-shot preparation. Therefore, permeation tubes generally require a continuous flow of matrix gas, even when they are not being used for calibration. When not in use, the flow should be vented. This type of system requires a modification of the typical analytical calibration standard strategy in which analysts make just

enough standard to perform a calibration.

Exponential dilution: In the exponential dilution method, a known amount of analyte or analyte mixture is injected into a flask of known volume and stirred well. A controlled flow of dilution gas travels through the flask continuously, mixes with the analyte, and sweeps it from the flask. The resulting concentration of analyte in the output flow begins with the ratio of the volume of analyte to the volume of the flask and continuously declines in an exponential manner. Figure 5 shows a typical experimental setup for exponential dilution.

Equation 8 describes the outlet concentration of analyte vapor:

$$C_i = C_0 e^{(-Ft/V)} \qquad [8]$$

where C_i is the outlet concentration of analyte i, C_0 is the initial concentration of analyte i in the exponential dilution flask, F is the volumetric flow rate of dilution gas, t is the time after sample introduction, and V is the volume of the flask or container.

The exponential dilution method is inexpensive and easy to understand. Each injection produces a full range of concentrations. The concentration at any given time is predictable using dilution theory. The technique can be used with a wide range of analytes, except for those with low volatility. For low analyte concentrations, the output of the flask can be diluted further with additional matrix gas.

Error sources are not well defined for exponential dilution. Some of the problems apparent in the exponential dilution method for preparation of calibration standards include surface sample losses, mechanical wear of the mixing device, and difficulty of accurately measuring the initial sample concentration caused by uneven mixing effects.

Comparison of Traceability in Calibration Methods

Static mixtures unquestionably are the most user-friendly form for preparing gas standards. Their preparation procedures are
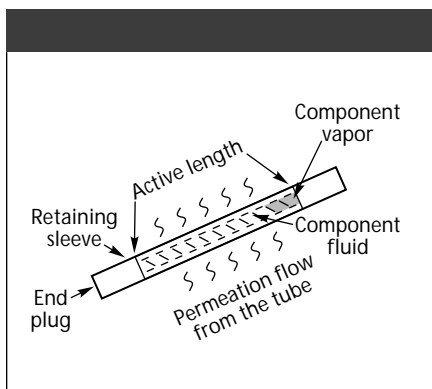
Figure 4: Schematic of a permeation tube.

straightforward, and the controlling variables — primarily mass measurements — can be traced easily to NIST. Two problems must be addressed with the preparation of every mixture: the stability of the mixture and the container's potential to contribute error to the mixture. Both of these considerations introduce undefined errors that destroy the value of traceability of the preparation process. Knowledge of the quantities of analyte and matrix placed into the container no longer implies knowledge of concentration actually delivered. Thus, static mixtures are traceable only by comparison to other traceable concentration standards.

Considering the aggressive nature of many analyte compounds, dynamic blending methods offer the path of least resistance to traceability. The strength of the dynamic approach is the avoidance of undefined error sources. If a known concentration is created through a traceable measurement, the fundamental variables are controlled, and the concentration is used immediately, then the unknown error sources of instability and container effects are avoided. The mixture is still traceable.

The dynamic blending approach has an additional side benefit. All methods, including direct use of a standard reference material, are subject to systematic delivery errors resulting from the interaction of an analyte with the materials used to transport the mixture from its source to the analytical system. Because traceable standards are precious, analysts tend to use them sparingly. When the delivery system is not totally inert to the mixture, this sparing use can result in significant undefined errors. With a continuous flow of mixture, allowing additional equilibration time in the transport system has little cost and tends to minimize the effect of those undefined errors.

As indicated in the sidebar, only direct gas blending, permeation tube techniques, and diffusion tube techniques are well

suited to achieving traceability. Next, we will compare and contrast these methods.

The concentration traceability of direct gas blending comes from the traceability of analyte and dilution gas flow measurements. The key problem in direct blending is that flow measurement traceability through NIST is available for very few gases, so most analytes have no direct traceability. Thus, the traceability for non-NIST gases requires users to relate analyte gas flow measurements to fundamental standards. This process is straightforward for well-behaved gases such as atmospheric gases and light hydrocarbons, but it is difficult to relate flow measurements to funda-

*The key advantage of **permeation tubes** is the range of compounds for which the method can be used.*

mental standards for reactive gases such as hydrochloric acid, hydrobromic acid, silanes, and arsines. Some analytical services for non-NIST organic compounds exist but are not well known to most users and are not equipped to handle a large customer base. In direct blending setups, it is virtually impossible to obtain traceability for the flow of vapor from compounds that exist as liquids at ambient conditions. In summary, traceability is available, but it must be dealt with on a case-by-case basis.

The concentration traceability for diffusion tube–based mixtures comes from the traceability of the diffusion tube emission rate and the dilution gas flow rate. The emission rate — the analyte flow rate — traceability derives from the traceability of weight-loss change measurement and elapsed time. The weight-loss method avoids the problem of needing a flow standard for analyte vapors. The diffusion tube method works well for liquids and subliming solids, but analysts must show that weight loss is actually caused by emission of analyte to establish traceability. The emission rate can vary depending upon user technique, so users should monitor weight loss continuously. The traceability of the dilution gas flow rate is straightforward. In summary, traceability of diffusion-based calibration mixtures is available for many analytes, but users must provide it.

The concentration traceability for permeation tube–based mixtures derives from the traceability of permeation tube emission rate and dilution gas flow rate. The emission rate — the analyte flow rate — comes from the traceability of weight-loss change measurements, elapsed time, and the operating temperature. Similar to the diffusion tube–based mixture, the weight-loss method avoids the problem of needing a flow standard for analyte vapors. For traceability, users must show that weight loss is actually the result of the emission of analyte from the permeation tube. The method works for liquid compressed gases such as hydrogen sulfide and sulfur dioxide and for liquids and subliming solids. It is unsatisfactory for fixed gases because the weight-loss method can't be used. After equilibrium is achieved for the permeation tubes, the emission rate is very stable and reproducible for long periods of time with stable analyte compounds. The traceability of the dilution gas flow rate is straightforward. In summary, permeation tubes provide traceability for most analytes.

Table I compares the methods for the preparation of traceable gaseous calibration mixtures. Each of the three methods has its advantages and disadvantages, and each should be considered based upon analyte type, required accuracy, and ease of traceability. Table II compares the expected accuracy of the three methods with assumptions.

Summary

The analysis of VOC vapors at parts-per-billion and even parts-per-trillion levels has become commonplace, both in laboratories and on-line. Concurrently, the adoption of documented quality assurance programs such as ISO 9000 has imposed requirements that all quality assurance measurements be calibrated against standards that are traceable to national or international standards. The problem is that at trace concentrations very few standards are available for VOC measurements.

Traceability can be achieved by preparing standards using a method in which the concentration is created as the direct result of fundamental measurements. In general, dynamic blending techniques are best suited for this purpose. Standards prepared from permeation tubes or diffusion tubes offer the most straightforward route to achieving traceability throughout a wide concentration range for most compounds. Commercial products are available for each of the approaches.

Table I: Comparison of dynamic methods for preparation of calibration standards

Property	Direct Blending	Diffusion Tube	Permeation Tube
Analyte	Gas	Liquid, solid	Gas, liquid, solid
Traceable variables	Analyte flow, diluent flow	Analyte weight loss, diluent flow, temperature	Analyte weight loss, diluent flow, temperature
Maximum concentration range*	100%	50 ppm	5000 ppm
Minimum concentration (one-step dilution)†	200 ppm	10 ppb	10 ppb
Minimum concentration (two-step dilution)†	40 ppb	10 ppt	10 ppt
Minimum analyte boiling point (°C)	N/A‡	40	N/A
Maximum analyte boiling point (°C)	20	~350	~280
Maximum pressure (psig)	~100	Near ambient	~100
Maximum analytes–mixture	6	90	240

*In 500 mL/min dilution flow.
†With 5000 mL/min maximum dilution flow per step.
‡N/A = not applicable.

Table II: Comparison of the expected accuracy of traceable calibration standard methods
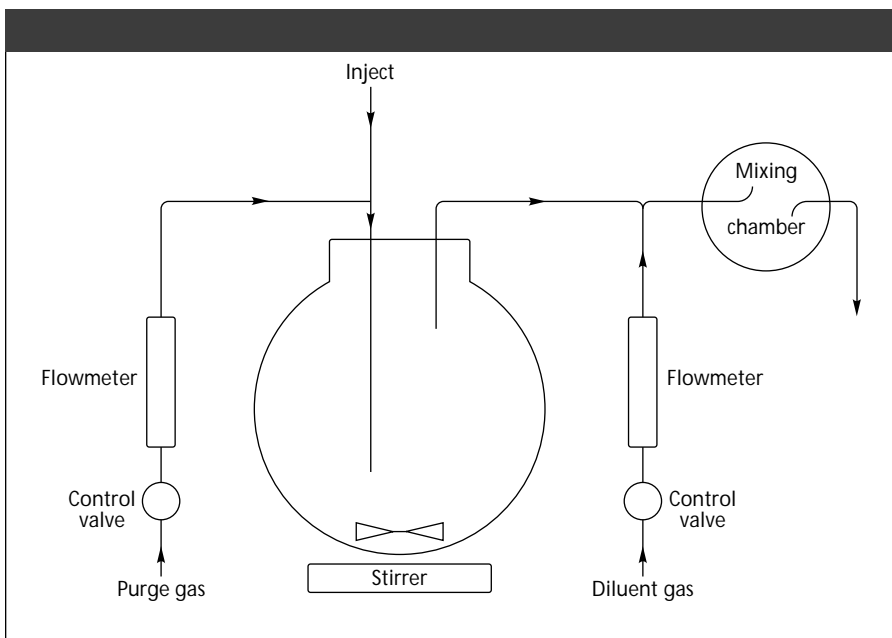
Technique	Concentration						
	5000 ppm	500 ppm	50 ppm	5 ppm	500 ppb	50 ppb	5 ppb
Direct blending*	±2	±2	±4	±4	±4	±4	±6
Direct blending†	±6	±6	±8	±8	±8	±8	±10
Diffusion	N/A	N/A	±5	±5	±5	±5	±7
Permeation*‡	±2	±2	±2	±2	±2	±2	±4
Permeation*§	±4	±4	±4	±4	±4	±4	±6

* Assumes flowmeter calibration with actual analyte gas to ±1% of measured value.
† Assumes analyte flowmeter calibrated to surrogate gas and flow value obtained by applying flow factor to measured value.
‡ Assumes emission rate measured in the generating apparatus at operating conditions.
§ Assumes typical commercial practice using factory-certified tubes.

Figure 5: Exponential dilution flask apparatus for preparation of standard calibration mixtures.

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