CHAPTER 6

SOURCES OF BIAS IN THE GAS ANALYZER

Chapter 6 Highlights

Gas Analyzer Problems

Problem		Corrective Actions	Page Refs
Name	**Description**		
General			
Interference Effects	The presence of other gas species throws off the measurement of the gas being monitored.	Change analysis technique. Measure concentration of interferent and correct for its presence. Scrub out the interfering species before analysis.	6-2–6-5
Analyzer Design	Features inherent in an analyzer's physical construction, electronic design, and analytical technique can be prone to producing measurement bias.	Choose analyzers wisely, considering bias-prone features. In QA/QC program, tailor preventative maintenance to design features that are bias prone.	6-7
Ambient Effects			
Temperature	If exposed to extreme temperatures, the analyzer may produce erroneous readings.	Temperature stabilize the analyzer. Measure temperature and compensate.	6-5, 6-6
Pressure	Changes in barometric pressure can introduce systematic error in spectroscopic systems where measurements are made from a sample cell.	Monitor pressure and mathematically compensate for pressure effects.	6-6, 6-7
Polluted/ Corrosive Atmosphere	Situations like plume downwash or flue gas exhausting into CEM shelter can produce systematic error or system failure.	Shelter or otherwise protect system. Filter ambient air.	6-6
Calibration			
Incorrect Gas Values	If the presumed and actual calibration gas concentrations differ significantly, biased measurements will result.	Replace or recertify gas. Find actual concentration. Recalculate effluent concentrations.	6-8
Inadequate/ Inconsistent Response Time	Bias can develop if operator or operating system does not allow adequate time for monitor to reach its asymptotic value.	Establish procedures that ensure consistently adequate time for monitor response.	6-9–6-11
System Calibration Obscuring Local Bias	Routine analyzer adjustments during full system calibration can mask local sources of bias.	Perform probe and local analyzer calibration checks in addition to system checks.	6-11, 6-12

CHAPTER 6

SOURCES OF BIAS IN THE GAS ANALYZER

6.1 INTRODUCTION

Bias in CEM systems can originate from the sampling system design, as discussed in Chapters 3 and 4, but it can also originate from the system analyzers. Analyzers used in CEM systems should be able to distinguish between the gas to be measured and the other components, or interferents, of the flue gas mixture. The ability of an analyzer to minimize the effects of interferents depends on the measurement principle employed or on the effectiveness of their removal before the gas is analyzed.

In addition to an instrument's capability for interference rejection, the construction and electronic design of an analyzer can also contribute to its measurement bias. Analyzer sensitivity to environmental factors, drift, response time, and noise, can all affect its performance. However, this performance can be evaluated in part by conducting calibration checks.

Calibration checks should be designed to provide an independent assessment of analyzer operation. In the United States, this independence is achieved by using audit gases traceable to NTIS standard reference materials (SRMs). However, internal references, such as sealed gas cells, filters, or reference spectra are also used to check analyzer calibration. Neither a calibration that uses an audit gas nor one based on an internal reference technique is completely independent of the CEM system, and both are subject to measurement biases that can be difficult to detect.

Four sources of analyzer bias, (1) interferences, (2) ambient effects, (3) design, and (4) calibration, can be avoided and usually resolved before, or during, CEM system certification. However, as the system ages, new calibration gases are purchased, or procedures are changed, biases caused by these factors may again enter into the system.

6.2 ANALYZER INTERFERENCE EFFECTS

It is not a trivial exercise to measure the concentration of SO_2, NO, CO_2, or O_2 in the mixture of gases emitted from combustion sources. Although it may be relatively easy to identify and measure the concentration of an isolated compound, it is the presence of other species that challenges analysis techniques. This challenge can be met in a number of ways, either by

1. Employing a technique that is specific to the compound being measured;
2. Measuring the concentration of all of the compounds, or the principal interfering compounds, and correcting for their presence; or
3. Removing the interfering species before analysis.

For continuously operating instruments, the first option is preferred although difficult to achieve. The analysis technique used is also dependent upon the CEM system design. The analysis options are reduced once the type of system is chosen (e.g., extractive, in-situ, dilution, hot-wet).

Typical interferences found in analyzers used for source monitoring are provided in Table 6–1.

Table 6–1. Typical Interferences Found in CEM System Analyzers

Technique	Typical Interferences
Infrared SO_2, NO, CO_2, CO	H_2O, CO_2, CO, Temperature
Luminescence SO_2, NO_x	CO_2, O_2, N_2, H_2O, Hydrocarbons (Quenching)
Ultraviolet (UV) SO_2 NO	 NO_2 SO_2
Electrochemical	Varies with cell EMFs
Electrocatalytic ZrO_2 cells for O_2	CO, Hydrocarbons
Paramagnetic O_2	NO

6.2.1 Instrument Design

Analyzer manufacturers first attempt to minimize these interferences through the instrument design. For example, in infrared analyzers, where overlapping O_2 and CO_2 spectral absorption bands are a problem, the technique of gas filter correlation can be used to minimize the interference of these gases. Since these spectra do not correlate with those of the pollutant molecules being measured, they merely attenuate the light in both the sample and reference modes of the instrument and do not lead to a bias. Also, the use of in-series Luft detectors has minimized the effect of H_2O and CO_2 interferences in the traditional nondispersive infrared analyzers.

Another example of instrument design being used to minimize interference effects can be seen in the steps taken in SO_2 fluorescence analyzers to reduce quenching effects caused by changes in percent level concentrations of O_2, CO_2, N_2, and H_2O. These changes have been found to cause errors from 5–15% in source-level fluorescence analyzers (Jahnke et al., 1976). The effects can be reduced by using shorter UV wavelengths, or by measuring the sample under reduced pressure. A better approach has been to dilute the sample with air to maintain a relatively constant background gas from which the SO_2 fluorescence radiation can be measured. Consequently these analyzers have been successfully employed in dilution systems.

6.2.2 Corrections

If the instrumentation cannot be refined to minimize interference effects, the next option is to measure the concentration of the interferent and mathematically correct for its influence. For example, this approach has been taken in second derivative SO_2 / NO analyzers, where the SO_2 interferes with the NO measurement. Both are measured and an electronic correction factor proportional to the SO_2 concentration is applied to the NO output. Also, in the electrocatalytic zirconium oxide O_2 analyzers, CO will react with the sampled oxygen to form CO_2 to deplete oxygen concentrations near the sensor. This problem only arises at high CO concentrations, but it can be resolved by measuring the CO concentration and correcting the O_2 output. In paramagnetic O_2 analyzers, NO, which is also paramagnetic, will interfere. Again, this is only a problem at high NO (percent) levels, but can be corrected if the NO is measured.

Other techniques can be applied in infrared and ultraviolet (UV) differential absorption analyzers. In these instruments, a complex spectral absorption curve is obtained of the flue gas. By using computer-maintained library spectra, spectra of the gaseous constituents at different concentrations can be matched until the sampled spectrum is reproduced. This and similar methods have been applied in Fourier transform infrared (FTIR) spectroscopic and differential optical absorption spectroscopic (DOAS) instru-ments.

In some cases, an analyzer can use an assumed correction factor for an interfering species rather than a measured one. For example, in a base-loaded power plant, CO_2 and H_2O concentrations are relatively stable, and rather than having to install and operate another analyzer to provide a correction factor, an assumed value established under normal operating conditions can be factored into the output. A modification of this method is to use a nomograph or computer algorithm that provides a variable correction factor based on flue gas concentrations (assumed or measured) or unit operating parameters. These corrections are only as good as the assumptions on which they are based and can lead to significant biases under atypical operating conditions. It is under these conditions, however, that accurate pollutant measurements are most desired.

Choosing analyzers for a CEM system requires some knowledge of how they operate and how interfering gases can affect the measurements. Unfortunately, vendor literature does not always provide detailed information on interference effects or methods used to correct for them. When data obtained from the system do not correlate with independent reference method tests or calculations made from plant operating information, further investigation may reveal the assumptions used in correcting the analyzer output.

6.2.3 Scrubbing

If it is not possible to account for interference through instrument design or corrections, the last option is to scrub out the interfering species from the sample gas. This was a traditional technique used in early CO nondispersive infra-red (NDIR) monitoring instrumentation.

Typically, both H_2O and CO_2 were scrubbed out in order to obtain an interference-free CO spectrum. Scrubbing is an adequate solution to the problem if the scrubbing materials are routinely replaced and if they do not remove any of the gas that is to be measured. However, when a major effluent component, such as CO_2, is removed, the sample volume must be adjusted to correct the apparent pollutant concentration measurements.

As discussed in Chapter 3, dilution air used in dilution probes or external dilution assemblies must be free of the compound to be measured. Gas scrubbers are routinely used for this purpose. Outside of this application, few scrubbing systems are used in utility CEM systems since analyzers are available today with enough discrimination to avoid using this last option.

6.3 AMBIENT EFFECTS

The environment in which an analyzer is located can also affect its performance. This is particularly true for in-situ analyzers but also holds true for extractive system analyzers. Ambient temperature, ambient pressure, vibration, and the composition of the ambient atmosphere, all can affect source monitoring instrumentation.

6.3.1 In-Situ Analyzers

In-situ analyzers can be subject to severe environmental conditions. These analyzers are either located on a stack or duct exposed to the atmosphere or in the annulus between the stack and stack liner. In either case, the instrument must be able to function properly through swings in temperature, variable humidity, and sometimes corrosive atmospheres.

Most in-situ analyzers are temperature stabilized and are usually covered with an aluminum or fiberglass hood. They are, however, not commonly air-conditioned, so if the temperature increases beyond specified limits, the instrument signal can drift dramatically or the instrument may simply malfunction. Locating a system on a metal stack, exposed to the sun, or in an annulus where elevated temperatures are commonplace should be avoided.

Effects of stack or duct vibration are not a common problem with in-situ analyzers, despite the frequent statements found in competitor trade literature. Vibrations found in stack installations are usually not at frequencies that will affect an instrument's performance. The manufacturer will otherwise make certain that optical and other system components are secured sufficiently to withstand the vibration encountered. It has been noted occasionally that circuit boards may loosen, but this will generally lead to major system fault rather than development of systematic errors in the measurement system.

In-situ analyzers are exposed to the ambient atmosphere and may be subject to plume downwash or acid liquids condensed from the exhaust gas. Manufacturers will generally seal the instrumentation in sturdy, cast aluminum housings to protect both optics and electronics from these atmospheres. However, continuing exposure to acid atmospheres will require more attention to maintenance and upkeep of the analyzer, particularly to the blower systems and clips and flanges that are prone to corrosion. Again, problems experienced here will tend not to lead to systematic errors, but rather to system failures requiring corrective action.

6.3.2 Extractive System Analyzers

Extractive system analyzers are commonly located in a CEM shelter that is temperature controlled. The assumption that shelter temperatures are stable may not always be true and can lead to drift problems in the system analyzers. The integrated circuits of the analyzers are temperature sensitive as they are in the in-situ systems. However, the extractive system analyzers, whether source level or ambient, may not be temperature stabilized if it is assumed that they will operate in a temperature controlled environment. In such cases, special care must be taken in sizing the heating and cooling system for the shelter. Inadequate systems can lead to cycling indoor temperatures that can ultimately cause unacceptable drift in the CEM systems.

EPA has not established temperature response performance criteria for CEM system analyzers, although temperature effects will contribute to the daily calibration error, which is limited to ±2.5% of span for system certification. Here, the ISO has established zero drift limits of less than or equal to ±2% and a span drift of less than or equal to ±4% of full scale for an ambient temperature change of ±10EC.

The ambient atmosphere must also be considered for CEM system shelters. Although analyzers are better protected in a shelter, introduction of pollutant gases into the shelter can lead to system biases. A problem can occur in some CO_2 infrared analyzers that are constructed with gaps between the measurement cell and the light source and detector. During a certification test, many people occupy the shelter, conducting tests, operations, or observations. With time, the CO_2 concentration in the shelter will increase. If the analyzer is used in a dilution system, the increased ambient levels of CO_2 will be detected by the analyzer to give abnormally high source CO_2 readings and invalidate the test. Similar problems may occur if the sampled flue gas is exhausted into the CEM shelter from the analyzer manifold or if the ambient air drawn into the shelter is contaminated from plume downwash.

Barometric pressure will also have an effect on extractive system analyzers where measurements are made from a sample cell. The pressure of these cells is generally maintained at ambient atmospheric pressure since the gas is sampled from a manifold that exhausts directly to the atmosphere. In many spectroscopic techniques, the measurements are sensitive to the sample cell pressure through pressure broadening of the measured spectra or by changing the density of the gas in the measurement cell. Consequently, to account for altitude, most analyzers incorporate adjustments that are set by the manufacturer after the installation location has been identified.

However, such altitude corrections do not account for day-to-day variation of **atmospheric** pressure. Changes in barometric pressure due to changing weather conditions can have a considerable effect on the analyzer measurements. The error introduced in the measurement is specific to the analyzer and should be obtained from the instrument manufacturer. Unfortunately, this information tends to be difficult to obtain, either because the manufacturer has not quantitatively determined the effects of barometric pressure or does not wish to acknowledge that the instrument is subject to such an effect. A number of manufacturers are now designing their systems to compensate for barometric pressure changes, or are offering the feature as an option. If it is desired to minimize analyzer drift, this option should be taken.

6.4 ANALYZER DESIGN

Both the physical construction and electronic design of a source monitoring analyzer are important in its operation. Although it is difficult to generalize here, it is noteworthy that today, state-of-the-art analyzers incorporating microcomputer circuitry and microprocessor firmware tend to be much more stable and drift- and noise-free than those constructed ten years ago. A number of analyzer manufacturers have not yet made this transition in technology. The older designs are still serviceable, however, and can meet today's certification requirements, including the bias test requirement.

Other design features are dependent upon the analysis technique used. For example, in differential absorption UV analyzers, lamp stability is an important factor in instrument operation. In infrared analyzers, detector sensitivity is important. These and other factors discussed above determine the quality of an instrument, its capability of meeting EPA certification requirements, and those design features most prone to systematic error. Taking into consideration bias-prone design features when choosing an analyzer can often head off future measurement problems. Careful consideration of analyzer design also allows operators to incorporate into their Quality Assurance and Control Programs preventative maintenance activities specifically tailored to design features that may be particularly susceptible to systematic error.

6.5 CALIBRATION

The periodic calibration of an analyzer is essential to its proper operation. The analyzer's electronics and optics, its response to environmental factors, and in some cases the effects of interferences can be checked through calibration.

Calibration is "the process of establishing the relationship between the output of a measurement process and a known input." In most instruments, it is too difficult to derive concentration values from first principles (e.g., using the Beer-Lambert law*) and in addition account for all of the other variables associated with the instrument's operation (such as electronic/optical design, environmental factors, and interferences discussed above). Instead, the instrument is calibrated so that it will give an accurate response to a known input.

The instrument manufacturer designs an analyzer to respond over a specified range of concentrations and programs in some relationship between the detector signal and the gas

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*Many electro-optical instruments dependent upon light-matter interactions incorporate a form of the Beer-Lambert law to obtain gas concentration values. The Beer-Lambert law is a theoretical expression that states that, for light having wavelengths that interact with the gas molecules, the transmission of the light through the gas will decrease exponentially as the concentration of the gas increases.

concentration. This may be a log-linear relationship for an infrared absorption analyzer, or a more complicated relationship, for example a second derivative function. The manufacturer then calibrates the instrument, injecting calibration gases of known concentration and obtaining a response. Instrument outputs are then adjusted to the known inputs to correct for variations of electronic signals, temperature effects, barometric effects, or possibly, interferences. Ideally, the analyzer will then provide an accurate response to the sampled gas after it is installed in the CEM system.

In the 40 CFR 75 CEM rules, the calibration of gas monitoring systems is required to be checked daily. The conditions under which a system was calibrated at the factory will be different than those at the installation location. Those conditions may also change daily. It has therefore been found necessary to check the calibration of a system daily.

6.5.1 Protocol 1 Gases

In the United States, a general policy has been set through the QA requirements of 40 CFR 60 and 40 CFR 75 that CEM systems be checked using calibration gases. More recently in Part 75, the requirement has gone further to specify that gases traceable to NIST SRMs are to be used. The traceability procedures established to do this are given as Protocol 1 (U.S. EPA, 1977a,b; 1993) and the required gases are known as Protocol 1 gases. In Europe, other methods of instrument calibration have been allowed and, as a consequence, European instruments may use sealed gas cells or reference spectra for self-calibration. Regardless of the adequacy of these methods, instruments used in the United States to meet Part 75 requirements must still provide some mechanism to be checked by Protocol 1 calibration gases.

6.5.2 Bias Due to Calibration Gases

Bias can be introduced into an analyzer's response if the calibration gas is not accurate. Protocol 1 gases are required to have an accuracy relative to an SRM of ±2%. The capabilities of gas manufacturers to provide accurate audit gases is periodically checked by EPA. These data are published and made available to the CEM user community.

Nevertheless, errors do sometime occur in the preparation of calibration gases. It is therefore prudent for the CEM system technician to cross-check newly purchased gases with other audit gases before they are used. Typically, a calibration gas cylinder is replaced before the cylinder pressure drops below 200 psi. Enough gas should be available to first calibrate the analyzer using the older cylinder gas, and then check the response of the analyzer to an injection of the new cylinder gas. If the response of the analyzer is within 2% of the certification value, the cylinder should be acceptable. If there is concern that the concentration of the gas in the older cylinder has degraded, further checks should be conducted using the audit gases reserved for quarterly linearity checks. Protocol gases provided by another plant or a source testing company could also serve this purpose. If the response differs by greater than 2% of the certified value, the cylinder should be returned to the cylinder gas manufacturer for reverification.

The use of multi-blend gases in dilution extractive systems has added another level of complexity into system calibration, as discussed in Chapter 3. The make-up gas used in the blend, and the average molecular weight of the blend must be considered when cross-checking gases. Since the critical flow rate of gas through a dilution orifice is dependent upon the average molecular weight of the gas, discrepancies will result if the average molecular weights of the compared gases are different (Miller, 1994; McGowan, 1994).

If it has been found that an incorrect cylinder gas value has been used to calibrate an analyzer or CEM system, the data obtained since that calibration will be biased. However, if the correct calibration gas concentration value is subsequently known, the true emission values can be determined. For example, if an SO_2 analyzer had been calibrated with a gas with an assumed value of 950 ppm and it was later found that the actual concentration was 900 ppm, measurements made by the analyzer would be too high.

The corrected concentration would be:

$$c_{corrected} = \frac{900}{950} c_{measured}$$

he measured concentrations would essentially need to be reduced since the original scale accounted too many parts per million for each part present in the sample.

6.5.3 Bias Due to System Response

Bias can occur in other ways in the calibration process. For example, consider that the response to a zero gas or a calibration gas is not always immediate, but will tend to an asymptotic value as shown in Figure 6–1.

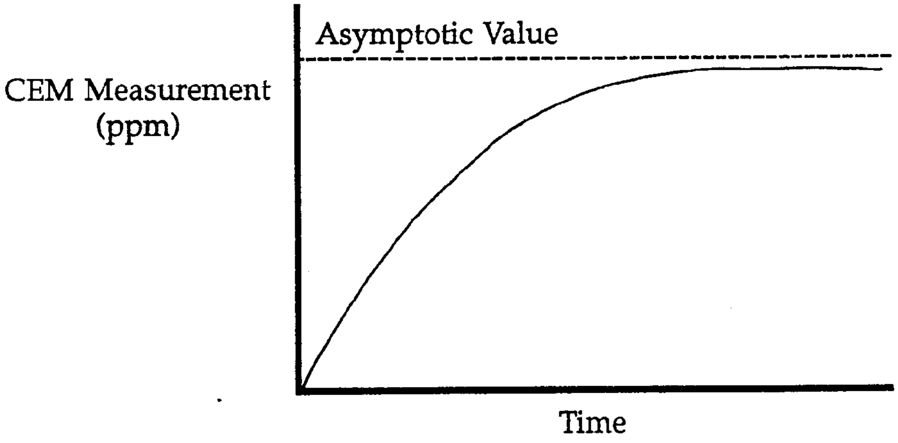

Figure 6–1. Asymptotic Calibration Check Response Curve

The time it takes to reach 95% of the asymptotic value is known as the response time. The system response time is due both to the time necessary for the gas to travel from the probe to the analyzer and the response time of the analyzer itself.

The analyzer response time is often dependent on an integration time, where the signal is averaged or integrated over a period that may range from seconds to minutes in order to obtain a more precise measurement. This period is fixed in many analyzers. However, in others, the integration period may be selected. The response time of many ambient air analyzers used in dilution systems may be on the order of several minutes, whereas the response time of an optical in-situ analyzer may be relatively rapid, on the order of seconds.

If a probe calibration is conducted (as is required in Part 75 for extractive systems and is necessary for dilution systems), the time necessary to flush out the probe and sampling line will enter into the "system" response. At low gas concentrations (e.g., less than 50 ppm), adsorption or desorption of gas on the walls of the transport tubing or sample cell may also delay the approach to the asymptotic value. In the case of adsorption, gas will be adsorbed on to the walls of the tubing, cell, and other surfaces until equilibrium is reached ("passivated"), after which time the cell concentration attains its final value. It may take 15–20 min to reach this value for some systems.

A bias problem develops when the technician or the automatic controller does not allow adequate time for the system to reach its asymptotic value during calibration checks. If the technician waits only 30 seconds before recording a reading on Day 1, but waits 60 seconds before recording a reading on Day 2, a noticeable change may occur in the instrument response. Also, when different technicians use different procedures for adjusting a system, significant biases can be introduced.

The solution to this problem is to adopt automated or consistent manual procedures for the daily calibration error check. In fact, EPA QA requirements specify that these procedures be written and followed. Calibration readings should be taken only after a specified period of time has elapsed. Calibration **adjustments** should be performed only after established QC limits have been exceeded (see Chapter 8). These control limits are also to be included in the written procedure. Frequent adjustments for variations of only a few ppm may serve only to adjust instrument noise and may not actually serve to improve data quality. In fact, the daily, automatic computer corrections for calibration drift performed by some systems may be doing nothing more than adjusting for system noise.

Many analyzers are sensitive to the pressure of the gas in the measurement cell or cavity. In the calibration mode, a high gas flow rate from the calibration gas cylinder can pressurize the cell and lead to subsequent sample measurement biases. In spectroscopic absorption instruments, the gas concentration measured is related to the number of molecules in the light path. If the pressure is increased, the density of the sample gas is increased. The number of the pollutant molecules in the light path increases correspondingly, even though their true concentration remains the same. The gas flow rate into the measurement cell must therefore be the same in both calibration and sampling modes. Since most gas manifolds are maintained to exhaust at a fixed pressure, the flow rates in both the calibration and sampling modes should be set so that this pressure is not exceeded.

6.5.4 System Calibration

In addition to analyzer calibration, one must also consider **system** calibration. The total response from a CEM system is through the "system" and not just the analyzer. As we have seen in Chapters 3 and 4, problems in the extractive system or in-situ monitor interface can also produce biased data. For this reason, the 40 CFR 75 rules require that daily "system" calibrations be conducted so that the combination of analyzer problems, extractive / interface system problems, and data acquisition system problems can be evaluated.

Routine analyzer adjustments performed during system calibration can hide analyzer measurement errors. These can be detected by performing both system and analyzer (local) calibration checks. For example, dilution systems must be calibrated ahead of the point where the dilution occurs. Since ambient air analyzers are used in dilution systems, the calibration gas (at Protocol 1 gas source level concentrations) must be diluted as is the sample gas. The dilution ratio of most dilution systems is determined approximately through selecting the size of the orifice or capillary and by setting an appropriate dilution air flow rate. The system is "tuned," however, by using calibration gas. Imprecise adjustment of the dilution flow rate and errors in the current absolute stack static pressure are all "calibrated out" with the analyzer. Variations in the dilution extractive system may be treated as analyzer drift, corrected by adjusting the span potentiometer or by adjusting the dilution air supply.

Using the analyzers to adjust for the variation of other system parameters can mask what is actually happening in the system. The combined system / analyzer calibration check may mask what is happening in each subsystem. For example, if the dilution probe controls begin to vary too far from their initial settings, there may not be enough adjustment capability in the analyzer potentiometers to bring the system into calibration. One technique that should be routinely applied to dilution systems is to perform a local analyzer calibration check to determine a baseline calibration setting. This baseline value can then be contrasted to the amount of adjustment needed to bring the whole dilution system into calibration. Since ambient air analyzers are used in the dilution systems, permeation tube systems may be necessary for the analyzer calibration check. More conveniently, a low-level concentration CO_2 gas (e.g., 3,000 ppm) could be used to assess the system (Gregoria, 1993).

A particularly aggravating problem can occur in time-shared dilution systems (Figure 6–2). In the system shown, a series of system calibrations would be performed, in turn, through each of the three dilution probes. First, for Unit 1, the analyzers are adjusted for the extractive system as well as analyzer variables. When calibrating the extractive system for Unit 2, the same set of analyzers are then adjusted for the variables associated with the dilution system of Unit 2; likewise for Unit 3. The same analyzers must then meet three separate sets of conditions. The system would then need to be designed to apply a different set of calibration conditions when each unit is being monitored. This could be done through the DAHS, but adds another layer of complexity to the system.

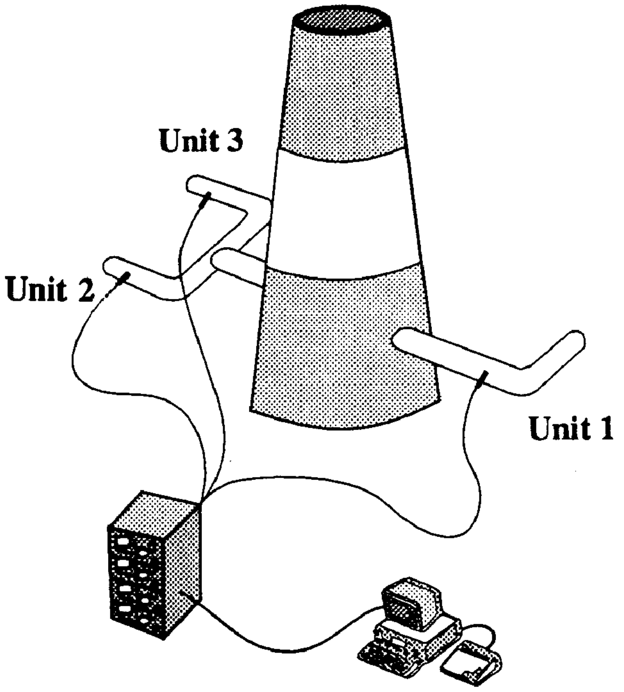

Figure 6–2. Problems in Calibrating Time-Shared Dilution Systems

6.6 SUMMARY

Factors that can cause errors in CEM system analyzers are summarized in the table on page 6-1. Although systematic in nature, many of these errors are variable, depending directly upon changing ambient conditions or levels of interferents. Due to this direct relationship, the resulting biases can in some cases be corrected if these underlying conditions are monitored and taken into account.

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