Simultaneous Calibration of Open-path and Conventional Point Monitors for Measuring Ambient Air Concentrations of Sulfur Dioxide, Ozone, and Nitrogen Dioxide

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ABSTRACT

A two-stage dilution system and an associated procedure to simultaneously calibrate both open-path (long-path) and conventional point air monitors have been used successfully during a comparison test study of open-path monitoring systems in Houston during August, 1993. Two open-path, differential optical absorption spectrometers (DOAS) were calibrated using standard concentrations up to 50 ppm in a 0.9 meter stainless steel optical cell connected to each DOAS analyzer via fiber optic cables. The calibration apparatus consisted of various flow controllers, flow meters, and mixing chambers to provide accurate dynamic flow dilutions; a high-concentration ozone generator; a suitable reaction chamber for gas phase titration (GPT) of NO to generate NO_2 standard concentrations; and an output manifold. High-concentration standards for SO_2 and NO were obtained by the primary dilution of nominal 1000-ppm, NIST-traceable concentration standards in compressed gas cylinders. NO_2 concentrations were generated by GPT from NO concentrations. The ambient-level concentration standards were provided by quantitative secondary dilution of the high concentration standards. Ozone standard concentrations were generated by the high-concentration ozone generator and assayed, after secondary dilution to ambient-level concentrations, by a commercially available UV photometric ozone analyzer used as a transfer standard.

INTRODUCTION

Instruments that measure atmospheric pollutants over an extended, linear, open path through the atmosphere offer several significant advantages relative to conventional air pollutant monitors (point analyzers), which extract an air sample from the atmosphere at a single point for analysis inside the analyzer. The primary advantages are much better spacial representation of a geographical area and better probability of capturing measurements of local sources under variable wind directions. Other advantages include greater siting flexibility, capability of monitoring areas or locations not readily accessible with conventional monitors, measurement of pollutants *in situ* without risk of pollutant alteration due to contact with surfaces and components in the inlet and analytical systems of

conventional monitors, and capability for monitoring multiple pollutants simultaneously with the same instrument.

Open-path, differential optical absorption spectrometer (DOAS) monitoring instruments have been used for several years in Europe to monitor O_3, SO_2, and NO_2 in the ambient air. Such instruments are now commercially available in configurations suitable for potential application in the United States by state and local air monitoring agencies. However, two things must happen before open-path instruments can be used in EPA-required state and local air monitoring stations (SLAMS): 1) Open-path analyzers must be formally designated by EPA as equivalent methods,[1] and 2) the EPA monitoring regulations[2] must be amended[3] to accommodate the use of open-path* pollutant measurements and to govern the application, siting, operation, and quality assurance of open-path analyzers. A critical part of the latter requirement is the quality assurance procedures associated with the operation of open-path analyzers, particularly calibration and assessment of accuracy and precision of open-path pollutant measurements.

In an effort to gather operational information and experience with open-path monitoring analyzers in support of the development of regulatory amendments, EPA carried out a field study of the technology in Baytown (Houston), Texas in August, 1993. This site, on property owned by the Exxon Corporation, was selected because of the availability of an existing, well-equipped, conventional air monitoring station; substantial concentrations of SO_2, NO_2, and O_3 typically recorded at the site; suitable siting for the installation of open-path monitoring instruments; and convenient site access and security. Since one objective of the study was to compare the performance and pollutant measurements of open-path and conventional analyzers, both types of analyzers were operated in the study. In addition to the existing conventional monitoring station, a second monitoring station housing conventional analyzers for SO_2, NO_2, and O_3 and two open-path DOAS analyzers were installed for the study. The DOAS instruments were manufactured by Opsis AB (Furulund, Sweden); one was owned by EPA and the other was provided for the study by Opsis AB. Both DOAS instruments operated with parallel paths, each 400 meters long.

Another objective of the study was to test the efficacy of the calibration technique recommended by the manufacturer and the audit and precision check procedures proposed by the manufacturer for incorporation into the quality assessment requirements of the monitoring regulation.[4] Because of the comparative aspects of the study, it was deemed important to calibrate both the conventional analyzers and the open-path instruments simultaneously, using the same calibration standards. Accordingly, a calibration system and associated calibration procedure for simultaneous calibration of both types of analyzers for SO_2, NO_2, and O_3 was assembled and used successfully during the field study.

CALIBRATION SYSTEM DESCRIPTION

The calibration technique prescribed by Opsis for its DOAS instruments utilizes an optical calibration cell having quartz end windows and gas ports through which pollutant gas concentration standards can be passed. The regular outdoor ultraviolet (UV) light transmitter and receiver used for monitoring are temporarily replaced during calibration by a local UV light source and two smaller receivers. These receivers are mounted to a calibration bench, which holds them and the calibration cell in optical alignment, as shown in Figure 1. One receiver is connected, via a fiber optic cable, to the DOAS analyzer in place of the fiber optic cable from the normal (outdoor)

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*It is actually the "long-path" nature of the pollutant measurement, *i. e.* the averaging of pollutant concentrations in the atmosphere over a path many meters long (path-integrated concentration), that is of concern with respect to the EPA monitoring regulations. Unfortunately, the term "long path monitoring" has been (tentatively) defined by A&WMA Technical Committee EM-6 in its Remote Sensing Glossary to apply to optical instruments having a *folded* optical path, either with the path in a closed cell or open to the atmosphere. In the later case (which should *logically* be categorized as an open-path analyzer) the actual monitoring path through the atmosphere is typically so short (less than 1 meter, for example) that EPA would consider the instrument to be a point analyzer for regulatory purposes. Committee EM-6 defines "open-path monitoring" in the extended path sense; therefore, "open-path" is used in this paper to be consistent with that definition (pending possible further refinement of the two definitions).

monitoring receiver. The other receiver is connected to the local light source via another fiber optic cable and thereby becomes, in effect, a light transmitter. The internal surfaces of the stainless steel calibration cell are chemically polished to minimize surface reaction or adsorption of the pollutant gases inside the cell, which could alter the standard concentrations.

The calibration apparatus used for the simultaneous calibration is shown schematically in Figure 2. The system was based primarily on two-stage, quantitative, dynamic dilution of NIST-traceable, high-concentration standards of NO and SO_2 from compressed gas cylinders. Small flow rates (10 to 100 cm^3/min) from either the NO or SO_2 standard gas cylinder were measured and controlled by flow controller 1. Zero air could also be metered by this flow controller to maintain identical flow conditions during periods when neither NO nor SO_2 was required. Zero air for the primary dilution was metered and controlled by flow controllers 2 and 3. As shown, this dilution flow was divided so that a portion of it passed through an ozone generator, which could be turned on or off electrically. These three flows then passed into reaction and mixing chambers (glass Kjeldahl-type mixing bulbs) and into the DOAS optical calibration cell. The flow controllers were accurately calibrated with a Gillian bubble flow standard, and the cell concentrations of NO or SO_2 were then calculated from the dilution ratio, as $C_{cell} = C_{std} \times F_1/(F_1 + F_2 + F_3)$. A bypass around the calibration cell was also provided to allow a convenient way to check for possible O_3 or NO losses in the cell by comparing the concentrations at the inlet and outlet of the cell.

The effluent gas from the calibration cell was then secondarily diluted by passing a portion of the flow (about 90 cm^3/min) through flow meter 5 and diluting it with a flow (about 9500 cm^3/min) of zero air measured and controlled by flow controller 4. This diluted gas standard was passed through another mixing device and into a vented manifold for connection of the conventional point analyzers under calibration. The final diluted pollutant concentration standard at this output manifold was calculated from the secondary dilution, as $C_{out} = C_{cell} \times F_5/(F_4 + F_5)$. Excess calibration gas effluent from the DOAS calibration cell was vented through a valve, which was necessary to create backpressure to force the gas through flow meter 5 and the remainder of the apparatus. This resulted in the pressurization of the calibration cell above atmospheric pressure and necessitated a corresponding pressure correction in the DOAS measurement of the calibration cell concentration. A manometer was connected at the outlet of the cell to measure the cell pressure for the pressure correction. Originally, a mass flow controller was tried in place of flow meter 5 to automatically regulate flow rate F_5 for the secondary dilution. However, the considerably higher backpressure required for proper flow controller operation greatly increased the potential for errors due to leaks in the system and created a potential hazard from increased pressure in the glass reaction and mixing chambers. Hence, flow rate F_5 had to be regulated manually via the needle valve on the cell vent.

Ozone standards were generated by a stable but uncalibrated, high-concentration ozone generator, then assayed, after quantitative dilution to ambient-level concentrations, by a NIST-traceable ozone transfer standard. The ozone generator utilized a mercury vapor lamp to generate ozone, similar to ozone generators for ambient-level concentrations. To get the much higher concentrations needed, the lamp was larger, and the air flow completely surrounded the lamp, which operated inside a chamber several centimeters in diameter. A mechanical sleeve that could be slid over part of the lamp was used to change the ozone concentration without changing the flow rate through the generator. Since the lamp was turned off when ozone was not needed, the generator required about 20 minutes after being turned on to provide a stable ozone concentration. The ozone concentration in the DOAS calibration cell was calculated as $C_{cell} = C_{out} \times (F_4 + F_5)/F_5$, where C_{out} is the ozone concentration in the output manifold as assayed by the ozone transfer standard.

NO_2 standards were generated by gas phase titration (GPT).[5],[6] An NO standard concentration was established at a secondary dilution output of about 400 to 450 ppb, as measured by the (previously calibrated) point NO analyzer. A small amount of the NO was converted to NO_2 by air oxidation due to the high concentrations and fairly long residence time in the system. However, the amount was usually small, of the order of a few ppb, as measured at the outlet manifold. The

ozone generator was then turned on to generate sufficient O_3 to titrate up to 80% - 85% of the NO to NO_2. After the system restabilized, the remaining NO concentration was determined by the NO point analyzer, and the NO_2 concentration was calculated as the difference between the original and final concentrations of NO, with some adjustment to account for the NO_2 present prior to addition of the O_3. As with O_3, the concentration of NO_2 in the calibration cell was calculated as, $C_{cell} = C_{out} \times (F_4 + F_5)/F_5$, where C_{out} was the NO_2 concentration determined in the output manifold.

CALIBRATION OF THE DOAS INSTRUMENTS

The particular calibration cell used during our work had a measured internal length of 0.900 meters, while the installed DOAS monitoring path length was 407 meters. To be equivalent to ambient concentrations, pollutant standard concentrations in the cell therefore would have to be higher by a factor of 407/0.900 or 452. Ambient air pollutant monitors for SO_2, NO_2, and O_3 are normally calibrated over a concentration range of 0 to 0.5 ppm; thus, calibration standard concentrations in the cell should be in the range of 0 to 226 ppm for an equivalent calibration range for the open-path instruments. In our simultaneous calibration apparatus, the standard concentrations obtainable were limited to about 50 ppm because of several constraints, including the ranges of the flow meters that were available for the apparatus, the maximum flow rate capacity of the zero air supply system, a minimum flow rate limit for the cell to maintain a reasonable flush time, and a practical limit on the dilution ratio of the secondary dilution. A 50 ppm cell concentration was equivalent to an ambient concentration of about 0.110 ppm, which covered most the ambient concentration levels measured during the study. To provide consistency in the calibration results for both point and open-path analyzers, all calibration cell concentrations were ultimately calculated as equivalent ambient concentrations (effective concentrations), using Equation (1):

$$C_{\mathit{eff}} = C_{\mathit{cell}} \frac{L_c}{L_m} \tag{1}$$

where: C_{eff} = Effective cell concentration, equivalent to an average ambient concentration over path length L_m, ppm
C_{cell} = Actual cell concentration, ppm
L_c = Calibration cell length, m
L_m = Opsis monitoring path length, m

The Opsis DOAS instrument is programmed to correct its open-path pollutant measurements for the ambient temperature and pressure. In our study, outdoor temperature was measured continuously with a temperature sensor, and the temperature corrections were based on this measured ambient temperature. Ambient (barometric) pressure was not monitored with a sensor, so the average barometric pressure for the site was manually entered into the Opsis software as a fixed value. During calibration, the temperature in the calibration cell, which was located indoors, was likely to be somewhat different than the outdoor ambient temperature, and the cell was slightly pressurized. These temperature and pressure differences could be handled in several ways, either automatically by the analyzer or manually, external to the analyzer. Choosing the former, we manually entered the cell temperature and pressure values into the analyzer software and reprogrammed the analyzer to use these special fixed values during the calibration rather than the average barometric pressure and the temperature readings obtained from the outdoor sensor. The reason for this choice was that the absorption cross section for SO_2 has a small temperature dependency, which the instrument takes into account in its measurement calculations. For O_3 and NO_2, which show no similar temperature dependency, we could have just as well elected to make the corrections manually, external to the analyzer. With the temperature and pressure corrections taken care of by the analyzer, the DOAS instrument readings of the calibration cell concentrations needed only to be converted from $\mu g/m^3$ units to ppm, using Equation (2):

$$C'_{rdg} = C_{rdg} \frac{273}{298} \frac{1}{F_{25}}, ppm \qquad (2)$$

where: C'_{rdg} = Measured concentration of the calibration cell contents, effective ppm
C_{rdg} = Cell concentration reading reported by the DOAS, $\mu g/m^3$ corrected to 0 °C and 760 mm Hg and assuming monitoring path length L_m
273/298 = Temperature adjustment from 0°C to 25°C
F_{25} = EPA units conversion factor at 25°C, $\mu g/m^3/ppm$;
$F_{25}(NO_2)=1880$; $F_{25}(O_3)=1960$; $F_{25}(SO_2)=2620$

The Opsis DOAS instrument reports all pollutant measurements in units of $\mu g/m^3$ corrected to 0°C and 760 mm Hg. In converting these units to ppm, the 273/298 term is included so that the familiar EPA unit conversion factors calculated at 25°C (F_{25}) can be used. (Alternatively, the unit conversion factors could be recalculated at 0°C and the 273/298 term eliminated.) For convenience during the actual calibration adjustment of the instrument, equation 2 can be rearranged and C'_{rdg} replaced with C_{eff} to calculate the correct reading to set the instrument, as in Equation (3):

$$C_{rdg} = C_{eff} \frac{298}{273} F_{25} \qquad (3)$$

CALIBRATION OF THE CONVENTIONAL POINT ANALYZERS

Calibration of the conventional point analyzers was straight forward, using the calculated standard concentrations at the output manifold. The O_3 analyzer was calibrated directly with a photometric primary ozone standard that had been recently intercompared with the NIST-traceable Standard Reference Photometer maintained at EPA's Research Triangle Park laboratory. The calibrated O_3 analyzer was then used as a transfer standard to assay the ozone concentrations generated and diluted by the calibration system.

CALIBRATION RESULTS

The results of the calibration of the various point and open-path analyzers used in the study (after any calibration adjustment) are summarized in Table 1. The calibrations of the open-path analyzers are given in terms of effective or equivalent ambient concentrations to make them readily comparable with the point analyzers. All results show adequate calibration, with relatively minor variations. Admittedly, the results of a one-time calibration of this nature would be expected to be quite good, and no information was obtained to establish the stability and reproducibility of the calibration system. Further testing of the apparatus will be required to determine those performance characteristics.

As some confirmation of the good calibrations of both the open-path and point analyzers, Figure 3 presents preliminary ambient ozone measurement data from the three monitors for August 20, 1993. A high O_3 peak occurred in the afternoon of that day, exercising the analyzers over a considerable range of ambient concentrations. Ozone is usually well mixed in the ground-level atmosphere, and the two types of O_3 analyzers would be expected to agree well despite the differences in the location between the two DOAS monitoring paths and the point analyzer inlet. Figure 3 confirms good agreement among all three analyzers on this day over the substantial range of concentrations, with minor discrepancies apparent only at the higher concentration levels. Complete evaluation of the ambient pollutant measurements obtained during the study is currently in progress, and the results of that evaluation will be reported subsequently.

CONCLUSIONS

This work demonstrated the feasibility of simultaneous calibration of both open-path and conventional point analyzers, using the same concentration working standards for both types of instruments. Such simultaneous calibration is advantageous in situations where the two types of monitoring instruments are under comparison. Although the two types of analyzers require calibration standards in concentration ranges differing by up to 2.5 orders of magnitude, a calibration apparatus using a two stage (double) dilution system was successfully assembled and used in the field study. An O_3 generator of relatively simple design and using a conventional UV lamp was shown to provide suitable O_3 calibration concentrations for open-path instruments. Ozone concentrations up to 50 ppm were successfully generated, passed through an optical cell with minimal losses, and quantitatively diluted for assay by a conventional O_3 transfer standard at ambient-level concentrations. These O_3 concentrations also were successfully used to titrate NO to generate accurately known standard concentrations of NO_2, using the same GPT technique widely used to generate NO_2 standard concentrations at ambient levels.

Calibration of the Opsis open-path DOAS air monitoring instrument presented only relatively minor problems. Special calibration equipment is required, including an optical calibration cell, calibration bench, two optical receivers, an auxiliary light source and power supply, additional fiber optic cables, and a high-concentration ozone generator. Most of this equipment is available from the instrument manufacturer. The high-concentration O_3 generator may have to be assembled using some custom-made components. Concentration standards up to several hundred ppm may be required, depending on the ratio of the installed monitoring path length to the calibration cell length. Conventional calibrators typically used for point analyzers may provide suitable dilution capability if the pollutant concentration standards are in an appropriate range, although the O_3 generators in such calibrators probably will not provide high enough O_3 concentrations for O_3 calibrations or for NO_2 calibrations using GPT. Calibration of open-path analyzers for NO_2 using dilution of a high-concentration NO_2 compressed gas standard may be a potentially advantageous alternative to GPT.

Techniques proposed for conducting accuracy audits and precision checks of open-path analyzers for purposes of data quality assessment are very similar to the calibration techniques discussed for calibration. In general, data quality assessment checks should test the entire monitoring system in a configuration as close to the normal monitoring configuration as possible. Therefore, one salient difference is the need to try to use the same outdoor light source (transmitter) and optical path that are used for normal monitoring, rather than the alternate, local light source. Two possibilities for using the normal monitoring light transmitter and receiver are potentially available. In one, the same calibration bench, cell, and two small receivers are used, as shown in Figure 1 for the calibration configuration. However, the small receiver operating as a transmitter would be connected to the outdoor monitoring receiver rather to than the local light source. Thus, this system would use the light from the normal light transmitter, receiver, and light path and would thereby test the entire open-path monitoring system. The other possibility is to mount a special optical cell directly onto the outdoor monitoring receiver, to remain in the optical path at all times. Using this type of cell, precision or zero and span checks could be automated, greatly reducing the need for visits to the open-path monitoring site. In both techniques, the test measurements would have to be corrected because they would include both the prevailing ambient pollutant concentrations as well as the test concentrations. There are other potential problems with these quality assessment techniques, and further study and evaluation are needed before they are ready for adoption into the EPA monitoring regulations or guidance.

analyzer and various calibration equipment needed for the study; Lee Ann Byrd, of EPA's Office of Air Quality Planning and Standards for administrative planning and implementation of the field study; and Avis Hines, AREAL, for intercomparison of our ozone photometric standard against the NIST-traceable Standard Reference Photometer.

DISCLAIMER

REFERENCES

1. U. S. Environmental Protection Agency, Title 40, Code of Federal Regulations, Part 53; "Ambient Air Monitoring Reference and Equivalent Methods."

2. U. S. Environmental Protection Agency, Title 40, Code of Federal Regulations, Part 58; "Ambient Air Quality Surveillance."

3. Proposed amendments to Title 40, Code of Federal Regulations, Part 58; publication pending. Copies of the current draft are available from the first author.

4. U. S. Environmental Protection Agency, Title 40, Code of Federal Regulations, Part 58, Appendixes A, "Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS)" and Appendix B, "Quality Assurance Requirements for Prevention of Significant Deterioration (PSD) Air Monitoring."

5. U. S. Environmental Protection Agency, Title 40, Code of Federal Regulations, Part 50, Appendix F; "Measurement Principle and Calibration Procedure for the Measurement of Nitrogen Dioxide in the Atmosphere (Gas Phase Chemiluminescence)."

6. Ellis, E. C.; *Technical Assistance Document for the Chemiluminescence Measurement of Nitrogen Dioxide*, EPA-600/4-75-003; U. S. Environmental Protection Agency; Research Triangle Park, NC, 1976.

Table 1. Calibration data for both point and open-path analyzers.

Analyzer	Calibration range*, ppm	No. points	Slope	Intercept	Correlation coefficient
Point analyzer, SO_2	0 - 0.500	6	0.980	0.002	0.99998
Open-path analyzer 1, SO_2	0 - 0.110	6	0.999	0.000	0.99997
Open-path analyzer 2, SO_2	0 - 0.110	6	0.997	0.000	0.99995
Point analyzer, O_3	0 - 0.500	7	1.004	0.000	1.00000
Open-path analyzer 1, O_3	0 - 0.110	7	0.997	0.001	0.99985
Open-path analyzer 2, O_3	0 - 0.110	7	0.989	0.000	0.99991
Point analyzer, NO	0 - 0.500	6	0.986	0.000	0.99998
Point analyzer, NO_2	0 - 0.500	5	0.998	0.000	0.99960
Open-path analyzer 1, NO_2	0 - 0.110	5	1.013	0.000	0.99966
Open-path analyzer 2, NO_2	0 - 0.110	5	1.002	0.000	0.99981

*Effective (equivalent) ambient range for the open-path analyzers.
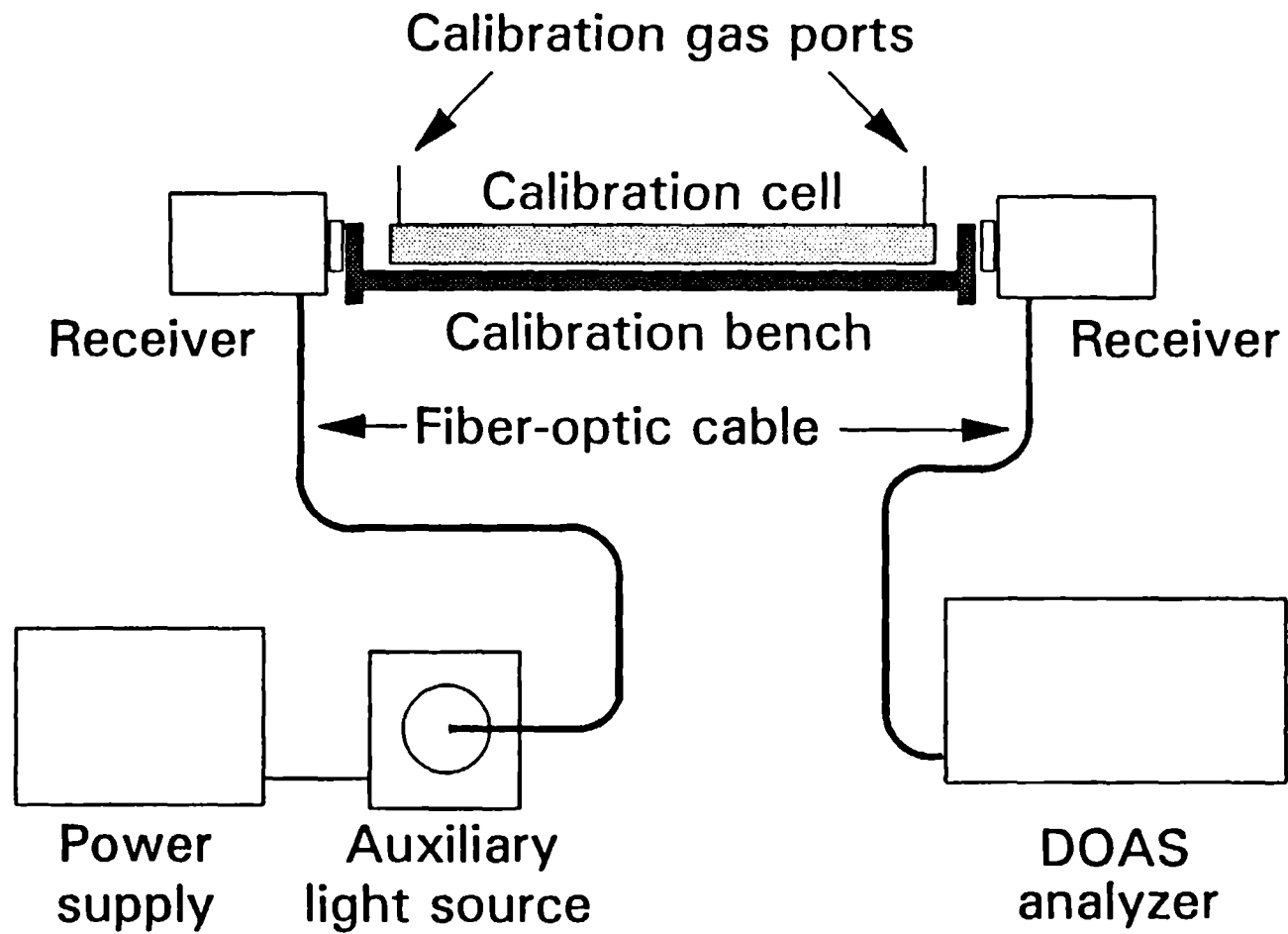
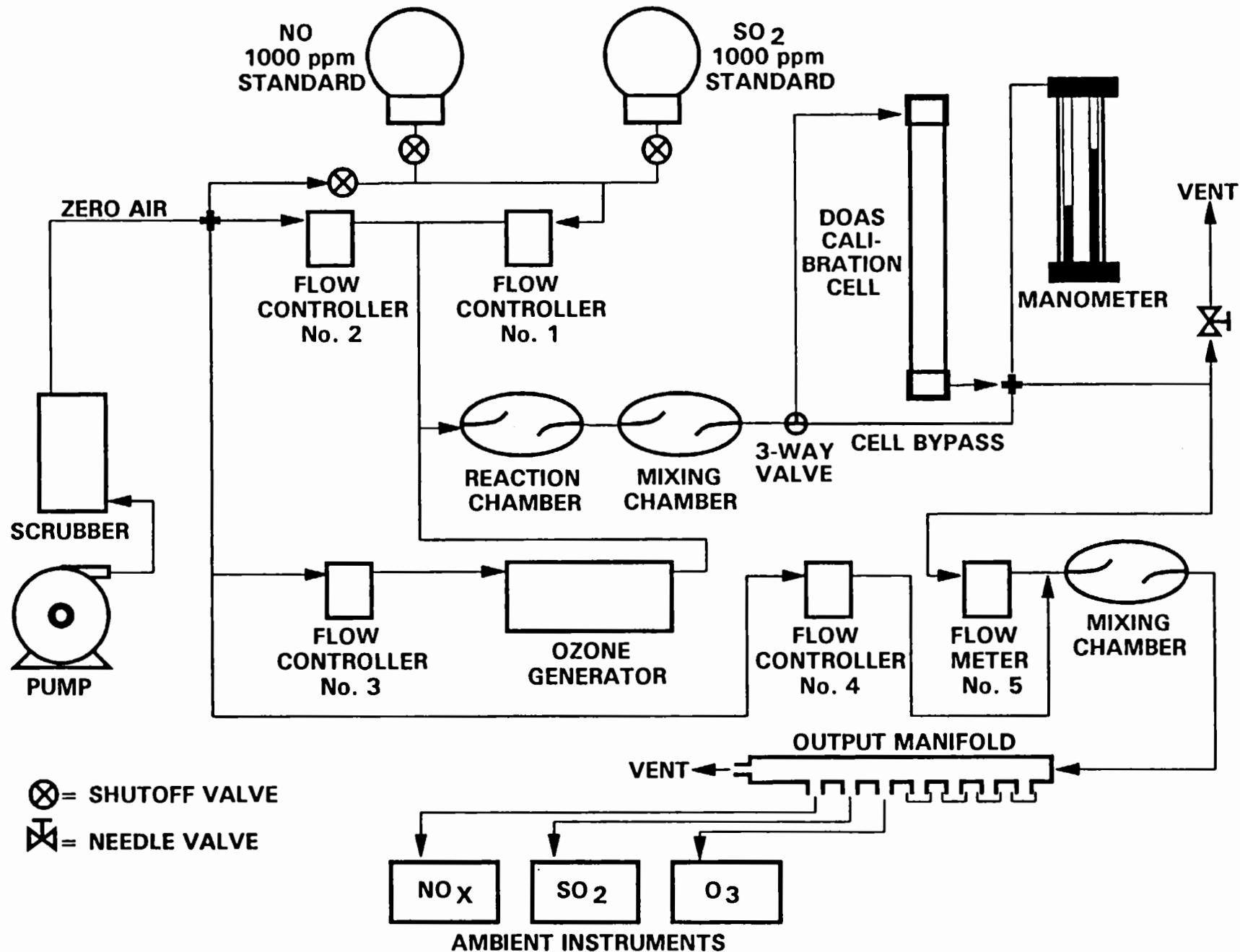Figure 1. DOAS calibration system.

Figure 2. Simultaneous calibration apparatus.

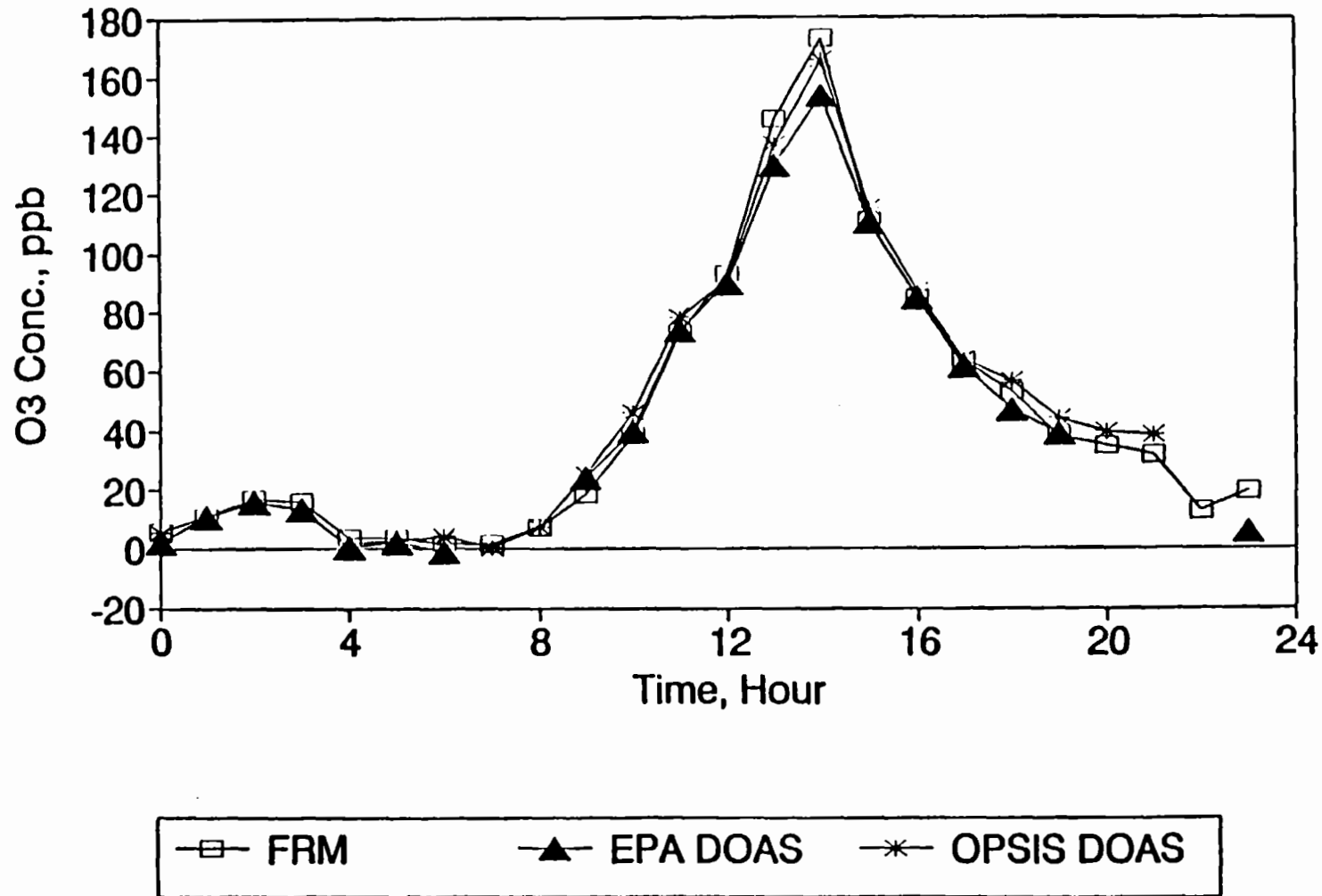# O3- Hourly Data- DOAS and FRM
August 20, 1993

Figure 3. Comparison of ambient O_3 measurements from point and open-path analyzers.

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16. ABSTRACT

A two-stage dilution system and an associated procedure to simultaneously calibrate both open-path (long-path) and conventional point air monitors have been used successfully during a comparison test study of open-path monitoring systems in Houston during August, 1993. Two open-path, differential optical absorption spectrometers (DOAS) were calibrated using standard concentrations up to 50 ppm in a 0.9 meter stainless steel optical cell connected to each DOAS analyzer via fiber optic cables. The calibration apparatus consisted of various flow controllers, flow meters, and mixing chambers to provide accurate dynamic flow dilutions; a high-concentration ozone generator; a suitable reaction chamber for gas phase titration (GPT) of NO to generate NO_2 standard concentrations; and an output manifold. High-concentration standards for SO_2 and NO were obtained by the primary dilution of nominal 1000-ppm, NIST-traceable concentration standards in compressed gas cylinders. NO_2 concentrations were generated by GPT from NO concentrations. The ambient-level concentration standards were provided by quantitative secondary dilution of the high concentration standards. Ozone standard concentrations were generated by the high-concentration ozone generator and assayed, after secondary dilution to ambient-level concentrations, by a commercially available UV photometric ozone analyzer used as a transfer standard.

17.	KEY WORDS AND DOCUMENT ANALYSIS		
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