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Standard Practice for General Ambient Air Analyzer Procedures[1]

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice is a general guide for ambient air analyzers used in determining air quality.

1.2 The actual method, or analyzer chosen, depends on the ultimate aim of the user: whether it is for regulatory compliance, process monitoring, or to alert the user of adverse trends. If the method or analyzer is to be used for federal or local compliance, it is recommended that the method published or referenced in the regulations be used in conjunction with this and other ASTM methods.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[2]

D 1357 Practice for Planning the Sampling of the Ambient Atmosphere[2]

D 3609 Practice for Calibration Techniques Using Permeation Tubes[2]

D 3670 Guide for Determination of Precision and Bias of Methods of Committee D22[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice other than those following, refer to Terminology D 1356.

3.1.2 *analyzer*—the instrumental equipment necessary to perform automatic analysis of ambient air through the use of physical and chemical properties and giving either cyclic or continuous output signal.

3.1.2.1 *analyzer system*—all sampling, analyzing, and readout instrumentation required to perform ambient air quality analysis automatically.

3.1.2.2 *sample system*—equipment necessary to provide the analyzer with a continuous representative sample.

3.1.2.3 *readout instrumentation*—output meters, recorder, or data acquisition system for monitoring analytical results.

3.1.3 *full scale*—the maximum measuring limit for a given range of an analyzer.

3.1.4 *interference*—an undesired output caused by a substance or substances other than the one being measured. The effect of interfering substance(s), on the measurement of interest, shall be expressed as: (±) percentage change of measurement compared with the molar amount of the interferent. If the interference is nonlinear, an algebraic expression should be developed (or curve plotted) to show this varying effect.

3.1.5 *lag time*—the time interval from a step change in the input concentration at the analyzer inlet to the first corresponding change in analyzer signal readout.

3.1.6 *linearity*—the maximum deviation between an actual analyzer reading and the reading predicted by a straight line drawn between upper and lower calibration points. This deviation is expressed as a percentage of full scale.

3.1.7 *minimum detection limit*—the smallest input concentration that can be determined as the concentration approaches zero.

3.1.8 *noise*—random deviations from a mean output not caused by sample concentration changes.

3.1.9 *operating humidity range of analyzer*—the range of ambient relative humidity of air surrounding the analyzer, over which the analyzer will meet all performance specifications.

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[1] This practice is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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[2] *Annual Book of ASTM Standards*, Vol 11.03.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

3.1.9.1 *operating humidity range of sample*— the range of ambient relative humidity of air which passes through the analyzer's sensing system, over which the monitor will meet all performance specifications.

3.1.10 *operational period*—the period of time over which the analyzer can be expected to operate unattended within specifications.

3.1.11 *operating temperature range of analyzer*—the range of ambient temperatures of air surrounding the analyzer, over which the monitor will meet all performance specifications.

3.1.11.1 *operating temperature range of sample*—the range of ambient temperatures of air, which passes through the analyzer's sensing system, over which the analyzer will meet all performance specifications.

3.1.12 *output*—a signal that is related to the measurement, and intended for connection to a readout or data acquisition device. Usually this is an electrical signal expressed as millivolts or milliamperes full scale at a given impedance.

3.1.13 *precision*—see Practice D 3670.

3.1.13.1 *repeatability*—a measure of the precision of the analyzer to repeat its results on independent introductions of the same sample at different time intervals. This is that difference between two such single instrument results, obtained during a stated time interval, that would be exceeded in the long run in only one case in twenty when the analyzer is operating normally.

3.1.13.2 *reproducibility*—a measure of the precision of different analyzers to repeat results on the same sample.

3.1.14 *range*—the concentration region between the minimum and maximum measurable limits.

3.1.15 *response time*—the time interval from a step change in the input concentration at the analyzer inlet to an output reading of 90 % of the ultimate reading.

3.1.16 *rise time*—response time minus lag time.

3.1.17 *span drift*—the change in analyzer output over a stated time period, usually 24 h of unadjusted continuous operation, when the input concentration is at a constant, stated upscale value. Span drift is usually expressed as a percentage change of full scale over a 24-h operational period.

3.1.18 *zero drift*—the change in analyzer output over a stated time period of unadjusted continuous operation when the input concentration is zero; usually expressed as a percentage change of full scale over a 24-h operational period.

4. Summary of Practice

4.1 A procedure for ambient air analyzer practices has been outlined. It presents definitions and terms, sampling information, calibration techniques, methods for validating results, and general comments related to ambient air analyzer methods of analysis. This is intended to be a common reference method which can be applied to all automatic analyzers in this category.

5. Significance and Use

5.1 The significance of this practice is adequately covered in Section 1.

6. Hazards

6.1 Each analyzer installation should be given a thorough safety engineering study.[5]

6.2 Electrically the analyzer system as well as the individual components shall meet all code requirements for the particular area classification.

6.2.1 All analyzers using 120-V, a-c, 60-Hz, 3-wire systems should observe proper polarity and should not use mechanical adapters for 2-wire outlets.

6.2.2 The neutral side of the power supply at the analyzer should be checked to see that it is at ground potential.

6.2.3 The analyzer's ground connection should be checked to earth ground for proper continuity.

6.2.4 Any analyzer containing electrically heated sections should have a temperature-limit device.

6.2.5 The analyzer, and any related electrical equipment (the system), should have a power cut-off switch, and a fuse or breaker, on the "hot" side of the line(s) of each device.

6.3 Full consideration must be given to safe disposal of the analyzer's spent samples and reagents.

6.4 Pressure relief valves, if applicable, shall be provided to protect both the analyzer and analyzer system.

6.5 Precautions should be taken when using cylinders containing gases or liquids under pressure. Helpful guidance may be obtained from Ref **(1), (2), (3), (4),** and **(5).**[6]

6.5.1 Gas cylinders must be fastened to a rigid structure and not exposed to direct sun light or heat.

6.5.2 Special safety precautions should be taken when using or storing combustible or toxic gases to ensure that the system is safe and free from leaks.

7. Installation of Analyzer System

7.1 Assure that information required for installation and operation of the analyzer system is supplied by the manufacturer.

7.2 Study operational data and design parameters furnished by the supplier before installation.

7.3 Review all sample requirements with the equipment supplier. The supplier must completely understand the application and work closely with the user and installer. It is absolutely necessary to define carefully all conditions of intended operation, components in the atmosphere to be analyzed, and expected variations in sample composition.

7.4 Choose materials of construction in contact with the ambient air sample to be analyzed to prevent reaction of materials with the sample, sorption of components from the sample, and entrance of contaminants through infusion or diffusion **(6, 7, 8, 9).**

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[5] The user, equipment supplier, and installer should be familiar with requirements of the National Electrical Code, any local applicable electrical code, U.L. Safety Codes, and the Occupational Safety & Health Standards (Federal Register, Vol 36, No. 105, Part II, May 29, 1971). Helpful guidance may also be obtained from API RP500, "Classification of Areas for Electrical Installations in Petroleum Refineries;" ISA RP12.1, "Electrical Instruments in Hazardous Atmospheres;" ISA RP12.2, "Intrinsically Safe and Nonincendive Electrical Instruments;" ISA RP12.4, "Instrument Purging for Reduction of Hazardous Area Classification;" and AP RP550, "Installation of Refinery Instruments and Control Systems, Part II."

[6] The boldface numbers in parentheses may be found in the Reference section at the end of this method.

7.4.1 Choose materials of construction and components of the analyzer system to withstand the environment in which it is installed.

7.4.2 Avoid the use of pipe-thread compounds in favor of polytetrafluorethylene tape.

7.5 Select the sampling point so as to provide a representative and measurable sample as close as possible to the sample system and analyzer (see Practice D 1357).

7.5.1 Provide a convenient access to the entire analyzer system.

7.5.2 Provide a necessary connection for introducing standard samples or withdrawing laboratory check samples immediately upstream of the analyzer sampling system.

7.6 Sample lines should be as short as practical.

7.6.1 Install the analyzer's exhaust so that no liquid or gas pressure buildup will occur. Provide proper venting, as far as possible from the sampling point.

7.7 After the installation has been completed, allow the analyzer to stabilize before testing performance specifications.

8. Calibration

8.1 One of the most important steps in analyzer operation is proper calibration of the instrument. Various calibration techniques may be used depending on the sample's physical or chemical property requiring measurement. Frequency of calibration depends largely on the application, degree of accuracy, and reliability expected. Perform calibration using spot samples (ambient) or a standard reference sample and utilize the analyzer adjustments as recommended by the manufacturer. Consult the supplier to determine the calibration procedure necessary for the particular analysis involved as preliminary instrument adjustments using zero and upscale standards may be necessary. Charts and calibration curves are essential and should be routinely verified.

8.1.1 In all cases, standard used for calibration purposes must be as representative as possible of the atmosphere to be analyzed, but cannot always contain all potential interfering substances.

8.2 *Spot Sample Calibration Method*— A sample is removed from the sampling line close to the analyzer inlet during a period when the sample flowing through the line is of uniform composition and the analyzer readout has reached an equilibrium value.

8.2.1 When this condition is reached, withdraw a sample from the inlet stream for analysis using the appropriate ASTM test method for the component of interest.

8.2.2 For most applications, a minimum of nine samples are required, and these shall be withdrawn each cycle for intermittent analyzers or for continuous analyzers after a stable response is achieved.

8.2.3 After each spot sample has been removed, record it as to time, sample number, date and corresponding analyzer readout. This equivalent readout is used in establishing a single calibration point.

8.2.4 Each spot sample must be analyzed in duplicate using the corresponding ASTM test method and the two results averaged. The standard deviation for the spot sample is calculated as the difference (larger value minus the smaller value) divided by $\sqrt{2}$. If this standard deviation exceeds the test method repeatability limit, *r*, (see Practice E 177) then that test average must be discarded. (This assumes that a repeatability limit has been determined for the test method and the laboratory conducting the test. This rejection criterion will discard 5 % of the spot sample results even if the test method is operating properly.)

8.2.5 Determine the amount of calibration offset by averaging the deviations, as shown in Table 1, and correct the analyzer readout accordingly. It may be necessary to review the manufacturer's recommended procedure for making calibration offset adjustments.

8.3 *Standard Sample Calibration Method*— Use a standard reference sample in accordance with the ASTM test method chosen, or by generating a known sample concentration, using NIST calibrated permeation tubes (see Practice D 3609).

8.3.1 A standard sample benchmark analysis is made by averaging the results of at least nine determinations using the corresponding ASTM test method. This average value is acceptable for benchmark analysis only if the corresponding standard deviation is lower in magnitude than the test method's repeatability limit, *r*, (see Practice E 177).

8.3.2 Check all operating parameters of the system in accordance with the instrument specifications and data for specific analysis. Allow sufficient time for the analyzer to reach equilibrium as indicated by a stable output.

8.3.3 Introduce the standard reference sample into the analyzer using the recommended instrument operational procedure. Activate the readout equipment.

8.3.4 After sufficient standard has been allowed to flow through the analyzer, adjust the readout to conform with the benchmark value. This establishes a single calibration point.

8.3.5 Continue introducing standard sample and record analysis after a stable response is achieved, or for each cycle if an intermittent analyzer is used, until repeatable data are recorded.

8.3.6 Discard any standard when any change in composition is detected (see Practice E 200).

9. Procedure

9.1 Begin sampling of the atmosphere.

9.2 Check all operating parameters in accordance with the application engineering data and method for specific analysis.

9.3 Observe the sample analysis as indicated by the readout equipment after the analyzer has been thoroughly purged with the sample.

TABLE 1 Typical Analyzer Calibration Using Spot Samples

Sample No.	Analyzer Results	ASTM Test Method Average	Difference
1	9.5	9.3	−0.2
2	9.5	9.4	−0.1
3	9.5	9.2	−0.3
4	9.7	9.5	−0.2
5	9.6	9.5	−0.1
6	9.5	9.3	−0.2
7	9.6	9.4	−0.2
8	9.4	9.2	−0.2
9	9.5	9.3	−0.2
		Calibration offset = (average difference)	−0.2

9.4 If it is desirable to validate the analyzer spot sample results, refer to the procedure given in Appendix X1.

9.5 After the analyzer is placed in service there is a continuing need to observe periodically that the original calibration remains valid. Achieve this by applying either the spot sample or standard sample technique used in calibration. The results logged over a period of time will indicate whether or not the analyzer remains within acceptable limits of calibration. Frequency with which these checks are required will be determined by the stability of the analyzer. If the record indicates frequent recalibration to be necessary, make a thorough investigation of the analyzer system to determine the cause of instability.

9.6 Successful operation of the analyzer system depends to a large extent on the amount of maintenance provided. Type of analyzer, complexity of the system, and condition of the sample stream usually determine the maintenance requirements.

10. Calculation

10.1 Each individual analyzer system, and ASTM test method chosen, determines the necessary calculations on the output signal. Most analyses are recorded as direct readouts based on instrument calibration. However, in some cases, the measurement sensitivity range is involved and scale factors are necessary to determine the final results. This is usually a simple multiplication step.

11. Report

11.1 Reports should include information on the analyzer system, calibration or validation used, and analysis of the sample over the time period involved. A report form is described in Appendix X1.

12. Precision

12.1 Preferably, each analyzer system method should include its own precision section based on cooperative test program results. This section would then incorporate the expected limit of deviation of test results from a determined value and be reported as repeatability and reproducibility.

13. Keywords

13.1 ambient air analyzers; ambient air quality

APPENDIX

X1. ANALYZER VALIDATION

X1.1 *Spot Sample Method:*

X1.1.1 Establish the validity of the spot sample data by comparing the analyzer results with the ASTM laboratory method results using the paired Student's "*t*" test.

$$t_c = \frac{(\bar{X}_i - \bar{X}_r)\sqrt{n}}{s} \tag{X1.1}$$

where:

X_i = instrument values for the ith sample,
X_r = individual benchmark values,
$\bar{X}_i$ = average instrument value,
$\bar{X}_r$ = average benchmark value,
n = number of spot samples corresponding to instrument and benchmark results, and

$$s = \sqrt{\frac{\Sigma(X_i - X_r)^2 - [\Sigma(X_i - X_r)^2]/n}{n - 1}} \tag{X1.2}$$

X1.1.2 Compare t_c with the values of "*t*" given in Table X1.1 for the number of degrees of freedom (df), $n - 1$, used in the calculation. If t_c is equal to or less than the tabulated value for "*t*," the instrument value can be considered valid. If t_c is greater than the tabulated value of "*t*," the instrument value differs from the benchmark value and the instrument results cannot be considered valid.

TABLE X1.1 Student's "*t*"Test for 95 % Confidence Level

Degrees of Freedom	"*t*"	Degrees of Freedom	"*t*"
1	12.706	17	2.110
2	4.303	18	2.101
3	3.182	19	2.093
4	2.776	20	2.086
5	2.571	21	2.080
6	2.447	22	2.074
7	2.365	23	2.069
8	2.306	24	2.064
9	2.262	25	2.060
10	2.228	30	2.042
11	2.201	40	2.021
12	2.179	50	2.008
13	2.160	60	2.000
14	2.145	120	1.980
15	2.131	200	1.960
16	2.120		

TABLE X1.2 REPORT FORM

Name of organization:

Date of analysis:

Time of analysis:

Barometric pressure:

Sample temperature:

Sample humidity:

Sample flow rate:

ASTM test method duplicated:

Analyzer type, model, serial No.:

Readout type, model, serial No.:

Analyzer range used:

Analyzer reading, uncorrected:

Analyzer corrections required, type and amount:

Analyzer reading, corrected:

Calibration validation used:

Date calibrated and validated:

REFERENCES

(**1**) *Safe Handling of Compressed Gases*, Pamphlet P-1, Compressed Gas Association, Inc., New York, NY.

(**2**) *Compressed Gases, Safe Practices*, Pamphlet No. 95, National Safety Council, Chicago, IL.

(**3**) "Handbook of Laboratory Safety," CRC Press, Boca Raton, FL, 1971.

(**4**) Sax, N. Irving, *Dangerous Properties of Industrial Materials*, 3rd Edition, 1968, Reinhold Book Corp., New York, NY.

(**5**) Matheson Gas Data Book—Sixth Edition, Matheson Gas Products, East Rutherford, NJ, 1980.

(**6**) Lebovits, Alexander, "Permeability of Polymers to Gases, Vapors and Liquids," *Modern Plastics*, March 1966, pp. 139–210.

(**7**) Hendrickson, E. R., "Air Sampling and Quantity Measurement," in Air Pollution (A. C. Stern, Ed.), Vol II, Academic Press, New York, NY, 1968, p. 23.

(**8**) Wilson, K. W., and Buchberg, H., *Industrial Engineering Chemical 50*, 1958, p. 1705.

(**9**) Baker, R. A., and Doerr, R. C., *Intl. Journal Air Pollution 2*, 1959, p. 142.

(**10**) Institute of Petroleum, Code of Practice—The Calibration, Verification and the Reporting of Analyzer Performance.