SUMMARY TABLE OF AQ/QC—Continued

Status	Process or element	QA/QC specification	Acceptance criteria	Checking frequency
			For wet-basis analyzers, keep sample above dew point at all times, by heating or dilution.	
S	Sample Extraction	Calibration valve material.	SS	Each test.
S	Sample Extraction	Sample pump material.	Inert to sample constituents	Each test.
S	Sample Extraction	Manifolding material	Inert to sample constituents	Each test.
S	Moisture Removal	Equipment efficiency	<5% target compound removal	Verified through system bias check.
S	Particulate Removal	Filter inertness	Pass system bias check	Each bias check.
M	Analyzer & Calibration Gas Performance.	Analyzer calibration error (of 3-point system calibration error for dilution systems).	Within ±2.0 percent of the calibration span of the analyzer for the low-, mid-, and high-level calibration gases.	Before initial run and after a failed system bias test or drift test.
			Alternative specification: ≤0.5 ppmv absolute difference.	
M	System Performance	System bias (or pre- and post-run 2-point system calibration error for dilution (Systems).	Within ±5.0% of the analyzer calibration span for low-sacle and upscale calibration gases.	Before and after each run.
			Alternative specification: ≤0.5 ppmv absolute difference.	
M	System Performance	System response time.	Determines minimum sampling time per point.	During initial sampling system bias test.
M	System Performance	Drift	≤3.0% of calibration span for low-level and mid- or high-level gases.	After each test run.
			Alternative specification: ≤0.5 ppmv absolute difference.	
M	System Performance	NO_2-NO conversion efficiency.	≥90% of certified test gas concentration	Before or after each test.
M	System Performance	Purge time	≥2 times system response time	Before starting the first run and when probe is removed from and re-inserted into the stack.
M	System Performance	Minimum sample time at each point.	Two times the system response time	Each sample point.
M	System Performance	Stable sample flow rate (surrogate for maintaining system response time).	Within 10% of flow rate established during system response time check.	Each run.
M	Sample Point Selection.	Stratification test	All points within: ±5% of mean for 1-point sampling. ±10% of mean for 3-point. Alternatively, all points within: ±0.5 ppm of mean for 1-point sampling. ±1.0 ppm of mean for 3-point sampling.	Prior to first run.
A	Multiple sample points simultaneously.	No. of openings in probe.	Multi-hole probe with verifiable constant flow through all holes within 10% of mean flow rate (requires Administrative approval for Part 75).	Each run.
M	Data Recording	Frequency	≤1 minute average	During run.
S	Data Parameters	Sample concentration range.	All 1-minute averages within calibration span.	Each run.
M	Date Parameters	Average concentration for the run.	Run average ≤calibration span	Each run.

S = Suggest.
M = Mandatory.
A = Alternative.
Agency.

10.0 Calibration and Standardization

What measurement system calibrations are required?

(1) The initial 3-point calibration error test as described in section 8.2.3 and the system bias (or system calibration error) checks described in section 8.2.5 are required and must meet the specifications in section 13 before you start the test. Make all necessary adjustments to calibrate the gas analyzer and data recorder. Then, after the test commences, the system bias or system calibration error checks described in section 8.5 are required before and after each run. Your analyzer must be calibrated for all species of NO_X that it detects. Analyzers that measure NO and NO_2 separately without using a converter must be calibrated with both NO and NO_2.

(2) You must include a copy of the manufacturer's certification of the calibration gases used in the testing as part of the test report. This certification must include the 13 documentation requirements in the EPA Traceability Protocol For Assay and Certification of Gaseous Calibration Standards, September 1997, as amended August 25, 1999. When Method 205 is used to produce diluted calibration gases, you must document that the specifications for the gas dilution system are met for the test. You must also include the date of the most recent dilution system calibration against flow standards and the name of the person or manufacturer who carried out the calibration in the test report.

11.0 Analytical Procedures

Because sample collection and analysis are performed together (see section 8), additional discussion of the analytical procedure is not necessary.

12.0 Calculations and Data Analysis

You must follow the procedures for calculations and data analysis listed in this section.

12.1 *Nomenclature.* The terms used in the equations are defined as follows:

ACE = Analyzer calibration error, percent of calibration span.

B_{WS} = Moisture content of sample gas as measured by Method 4 or other approved method, percent/100.

C_{Avg} = Average unadjusted gas concentration indicated by data recorder for the test run, ppmv.

C_D = Pollutant concentration adjusted to dry conditions, ppmv.

C_{Dir} = Measured concentration of a calibration gas (low, mid, or high) when introduced in direct calibration mode, ppmv.

C_{Gas} = Average effluent gas concentration adjusted for bias, ppmv.

C_M = Average of initial and final system calibration bias (or 2-point system calibration error) check responses for the upscale calibration gas, ppmv.

C_{MA} = Actual concentration of the upscale calibration gas, ppmv.

C_{Native} = NO_X concentration in the stack gas as calculated in section 12.6, ppmv.

C_O = Average of the initial and final system calibration bias (or 2-point system calibration error) check responses from the low-level (or zero) calibration gas, ppmv.

C_{OA} = Actual concentration of the low-level calibration gas, ppmv.

C_S = Measured concentration of a calibration gas (low, mid, or high) when introduced in system calibration mode, ppmv.

C_{SS} = Concentration of NO_X measured in the spiked sample, ppmv.

C_{Spike} = Concentration of NO_X in the undiluted spike gas, ppmv.

C_{Calc} = Calculated concentration of NO_X in the spike gas diluted in the sample, ppmv.

C_V = Manufacturer certified concentration of a calibration gas (low, mid, or high), ppmv.

C_W = Pollutant concentration measured under moist sample conditions, wet basis, ppmv.

CS = Calibration span, ppmv.

D = Drift assessment, percent of calibration span.

DF = Dilution system dilution factor or spike gas dilution factor, dimensionless.

Eff_{NO2} = NO_2 to NO converter efficiency, percent.

NO_{XCorr} = The NO_X concentration corrected for the converter efficiency, ppmv.

NOX_{Final} = The final NO_X concentration observed during the converter efficiency test in section 16.2.2, ppmv.

NO_{XPeak} = The highest NO_X concentration observed during the converter efficiency test in section 16.2.2, ppmv.

Q_{Spike} = Flow rate of spike gas introduced in system calibration mode, L/min.

Q_{Total} = Total sample flow rate during the spike test, L/min.

R = Spike recovery, percent.

SB = System bias, percent of calibration span.

SB_i = Pre-run system bias, percent of calibration span.

SB_{final} = Post-run system bias, percent of calibration span.

SCE = System calibration error, percent of calibration span.

SCE_i = Pre-run system calibration error, percent of calibration span.

SCE_{Final} = Post-run system calibration error, percent of calibration span.

12.2 Analyzer Calibration Error. For non-dilution systems, use Equation 7E–1 to calculate the analyzer calibration error for the low-, mid-, and high-level calibration gases.

$$ACE = \frac{C_{Dir} - C_V}{CS} \times 100 \qquad \text{Eq. 7E-1}$$

12.3 System Bias. For non-dilution systems, use Equation 7E–2 to calculate the system bias separately for the low-level and upscale calibration gases.

$$SB = \frac{C_S - C_{Dir}}{CS} \times 100 \qquad \text{Eq. 7E-2}$$

12.4 System Calibration Error. Use Equation 7E–3 to calculate the system calibration error for dilution systems. Equation 7E–3 applies to both the initial 3-point system calibration error test and the subsequent 2-point calibration error checks between test runs. In this equation, the term "C_s" refers to the diluted calibration gas concentration measured by the analyzer.

$$SCE = \frac{(C_S \quad DF) - C_V}{CS} \times 100 \qquad \text{Eq. 7E-3}$$

12.5 Drift Assessment. Use Equation 7E–4 to separately calculate the low-level and upscale drift over each test run. For dilution systems, replace "SB_{final}" and "SB_i" with "SCE_{final}" and "SCE_i", respectively, to calculate and evaluate drift.

$$D = |SB_{final} - SB_i| \qquad \text{Eq. 7E-4}$$

12.6 Effluent Gas Concentration. For each test run, calculate C_{avg}, the arithmetic average of all valid NO_X concentration values (*e.g.*, 1-minute averages). Then adjust the value of C_{avg} for bias using Equation 7E–5a if you use a non-zero gas as your low-level calibration gas, or Equation 7E–5b if you use a zero gas as your low-level calibration gas.

$$C_{Gas} = \left(C_{Avg} - C_M\right)\frac{C_{MA} - C_{OA}}{C_M - C_O} + C_{MA} \qquad \text{Eq. 7E-5a}$$

$$C_{Gas} = \left(C_{Avg} - C_O\right)\frac{C_{MA}}{C_M - C_O} \qquad \text{Eq. 7E-5b}$$

12.7 NO_2—NO Conversion Efficiency. If the NO_X converter efficiency test described in section 8.2.4.1 is performed, calculate the efficiency using Equation 7E–7.

$$Eff_{NO2} = \frac{C_{Dir}}{C_V} \times 100 \qquad \text{Eq. 7E-7}$$

12.8 NO_2—NO Conversion Efficiency Correction. If desired, calculate the total NO_X concentration with a correction for converter efficiency using Equation 7E–8.

$$NO_{x\,Corr} = NO + \left(\frac{(NO_X - NO)}{Eff_{NO_2}} x\, 100\right) \qquad \text{Eq. 7E-8}$$

12.9 Alternative NO_2 Converter Efficiency. If the alternative procedure of section 16.2.2 is used, determine the NO_X concentration decrease from NO_{XPeak} after the minimum 30-minute test interval using Equation 7E–9.

This decrease from NO_{XPeak} must meet the requirement in section 13.5 for the converter to be acceptable.

$$\% \, Decrease = \frac{NO_{XPeak} - NO_{XFinal}}{NO_{XPeak}} \times 100 \qquad \text{Eq. 7E-9}$$

12.10 Moisture Correction. Use Equation 7E–10 if your measurements need to be corrected to a dry basis.

$$C_D = \frac{C_W}{1 - B_{WS}} \qquad \text{Eq. 7E-10}$$

12.11 Calculated Spike Gas Concentration and Spike Recovery for the Example Alternative Dynamic Spiking Procedure in section 16.1.3. Use Equation 7E–11 to determine the calculated spike gas concentration. Use Equation 7E–12 to calculate the spike recovery.

$$C_{Calc} = \frac{\left(C_{Spike}\right)\left(Q_{Spike}\right)}{Q_{Total}} \qquad \text{Eq. 7E-11}$$

$$R = \frac{DF\left(C_{ss} - C_{native}\right) + C_{native}}{C_{Spike}} \times 100 \qquad \text{Eq. } 7E\text{-}12$$

13.0 Method Performance

13.1 Calibration Error. This specification is applicable to both the analyzer calibration error and the 3-point system calibration error tests described in section 8.2.3. At each calibration gas level (low, mid, and high) the calibration error must either be within ±2.0 percent of the calibration span. Alternatively, the results are acceptable if $|C_{dir} - C_v|$ or $|C_s - C_v|$ (as applicable) is ≤0.5 ppmv.

13.2 System Bias. This specification is applicable to both the system bias and 2-point system calibration error tests described in section 8.2.5 and 8.5. The pre- and post-run system bias (or system calibration error) must be within ±5.0 percent of the calibration span for the low-level and upscale calibration gases. Alternatively, the results are acceptable if $| C_s - C_{dir} |$ is ≤0.5 ppmv or if $| C_s - C_v |$ is ≤0.5 ppmv (as applicable).

13.3 Drift. For each run, the low-level and upscale drift must be less than or equal to 3.0 percent of the calibration span. The drift is also acceptable if the pre- and post-run bias (or the pre- and post-run system calibration error) responses do not differ by more than 0.5 ppmv at each gas concentration (i.e. $| C_{s\ post\text{-}run} - C_{s\ pre\text{-}run} |$ ≤0.5 ppmv).

13.4 Interference Check. The total interference response (i.e., the sum of the interference responses of all tested gaseous components) must not be greater than 2.50 percent of the calibration span for the analyzer tested. In summing the interferences, use the larger of the absolute values obtained for the interferent tested with and without the pollutant present. The results are also acceptable if the sum of the responses does not exceed 0.5 ppmv for a calibration span of 5 to 10 ppmv, or 0.2 ppmv for a calibration span <5 ppmv.

13.5 NO_2 to NO Conversion Efficiency Test (as applicable). The NO_2 to NO conversion efficiency, calculated according to Equation 7E–7, must be greater than or equal to 90 percent. The alternative conversion efficiency check, described in section 16.2.2 and calculated according to Equation 7E–9, must not result in a decrease from NO_{XPeak} by more than 2.0 percent.

13.6 Alternative Dynamic Spike Procedure. Recoveries of both pre-test spikes and post-test spikes must be within 100 ±10 percent. If the absolute difference between the calculated spike value and measured spike value is equal to or less than 0.20 ppmv, then the requirements of the ADSC are met.

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 Dynamic Spike Procedure. Except for applications under part 75 of this chapter, you may use a dynamic spiking procedure to validate your test data for a specific test matrix in place of the interference check and pre- and post-run system bias checks. For part 75 applications, use of this procedure is subject to the approval of the Administrator.

Best results are obtained for this procedure when source emissions are steady and not varying. Fluctuating emissions may render this alternative procedure difficult to pass. To use this alternative, you must meet the following requirements.

16.1.1 Procedure Documentation. You must detail the procedure you followed in the test report, including how the spike was measured, added, verified during the run, and calculated after the test.

16.1.2 Spiking Procedure Requirements. The spikes must be prepared from EPA Traceability Protocol gases. Your procedure must be designed to spike field samples at two target levels both before and after the test. Your target spike levels should bracket the average sample NO_X concentrations. The higher target concentration must be less than the calibration span. You must collect at least 5 data points for each target concentration. The spiking procedure must be performed before the first run and repeated after the last run of the test program.

16.1.3 Example Spiking Procedure. Determine the NO concentration needed to generate concentrations that are 50 and 150 percent of the anticipated NO_X concentration in the stack at the total sampling flow rate while keeping the spike flow rate at or below 10 percent of this total. Use a mass flow meter (accurate within 2.0 percent) to generate these NO spike gas concentrations at a constant flow rate. Use Equation 7E–11 in section 12.11 to determine the calculated spike concentration in the collected sample.

(1) Prepare the measurement system and conduct the analyzer calibration error test as described in sections 8.2.2 and 8.2.3. Following the sampling procedures in section 8.1, determine the stack NO_X concentration and use this concentration as the average stack concentration (C_{avg}) for the first spike level, or if desired, for both pre-test spike levels. Introduce the first level spike gas into the system in system calibration mode and begin sample collection. Wait for at least two times the system response time before measuring the spiked sample concentration. Then record at least five successive 1-minute averages of the spiked sample gas. Monitor the spike gas flow rate and maintain at the determined addition rate. Average the five 1-minute averages and determine the spike recovery using Equation 7E–12. Repeat this procedure for the other pre-test spike level. The recovery at each level must be within the limits in section 13.6 before proceeding with the test.

(2) Conduct the number of runs required for the test. Then repeat the above procedure for the post-test spike evaluation. The last run of the test may serve as the average stack concentration for the post-test spike test calculations. The results of the post-test spikes must meet the limits in section 13.6.

16.2 Alternative NO_2 to NO Conversion Efficiency Procedures. You may use either of the following procedures to determine converter efficiency in place of the procedure in section 8.2.4.1.

16.2.1 The procedure for determining conversion efficiency using NO in 40 CFR 86.123–78.

16.2.2 Bag Procedure. Perform the analyzer calibration error test to document the calibration (both NO and NO_X modes, as applicable). Fill a Tedlar or equivalent bag approximately half full with either ambient air, pure oxygen, or an oxygen standard gas with at least 19.5 percent by volume oxygen content. Fill the remainder of the bag with mid- to high-level NO in N_2 (or other appropriate concentration) calibration gas. (Note that the concentration of the NO standard should be sufficiently high enough for the diluted concentration to be easily and accurately measured on the scale used. The size of the bag should be large enough to accommodate the procedure and time required. Verify through the manufacturer that the Tedlar alternative is suitable for NO and make this verifed information available for inspection.)

(1) Immediately attach the bag to the inlet of the NO_X analyzer (or external converter if used). In the case of a dilution-system, introduce the gas at a point upstream of the dilution assembly. Measure the NO_X concentration for a period of 30 minutes. If the NO_X concentration drops more than 2 percent absolute from the peak value observed, then the NO_2 converter has failed to meet the criteria of this test. Take corrective action. The highest NO_X value observed is considered to be NO_{XPeak}. The final NO_X value observed is considered to be NO_{Xfinal}.

(2) [Reserved]

16.3 Manufacturer's Stability Test. A manufacturer's stability test is required for all analyzers that routinely measure emissions below 20 ppmv and is optional but recommended for other analyzers. This test evaluates each analyzer model by subjecting it to the tests listed in Table 7E–5 following procedures similar to those in 40 CFR 53.23 for thermal stability and insensitivity to supply voltage variations. If the analyzer will be used under temperature conditions that are outside the test conditions in Table B–4 of Part 53.23, alternative test temperatures that better reflect the analyzer field environment should be used. Alternative procedures or documentation that establish the analyzer's stability over the appropriate line voltages and temperatures are acceptable.

17.0 References

1. "ERA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards" September 1997 as amended, ERA–600/R–97/121.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

Figure 7E-1. Measurement System

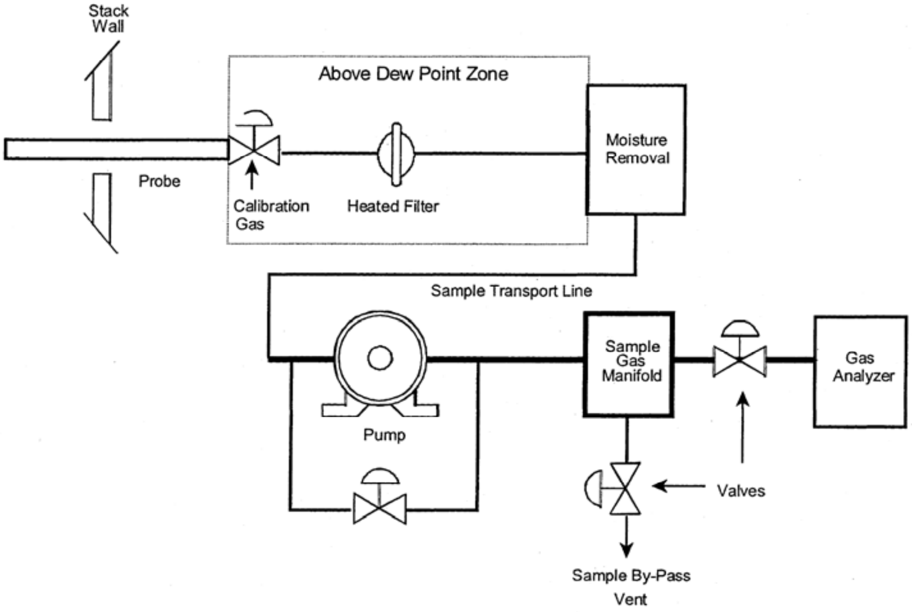
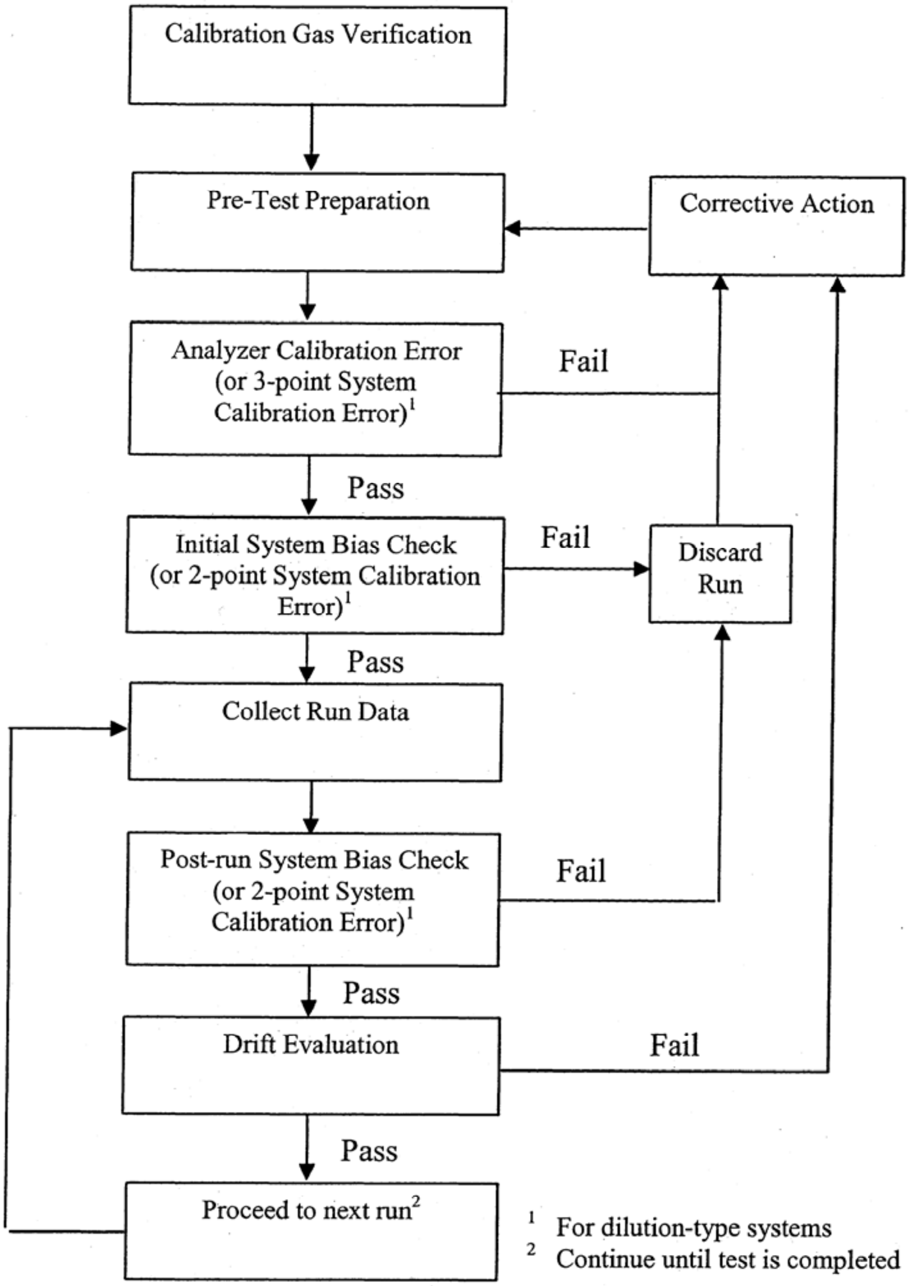


Figure 7E-2. Testing Flow Chart



[1] For dilution-type systems
[2] Continue until test is completed

Table 7E-1 - Analyzer (or System) Calibration Error Data

Source Identification: ____________
Test personnel: ____________________
Date: ____________________________
Time: ____________________________

Analyzer[1] or System[2] calibration error data for sampling runs: ______________________
Analyzer Model No. ____________________
Serial No. _____________________________
Calibration Span (CS):

	Manufacturer Certified Cylinder Value (indicate units) A	Analyzer calibration response (indicate units) B	Absolute difference (indicate units) A-B	Calibration Error (percent of calibration span) $\frac{A-B}{CS} \times 100$
Low-level (or zero) calibration gas	....	....	....	....
Mid-level calibration gas	....	....	....	....
High-level calibration gas	....	....	....	....

[1] Refers to data from the analyzer calibration error test of a non-dilution system.
[2] Refers to data from a 3-point system calibration error test of a dilution system.

Table 7E-2 - System Bias (or System Calibration Error) and Drift Data

Source Identification: ____________________ Run Number: ___________
Test personnel: ___________________________ Calibration Span: _________
Date: ___________________________________ ____ Response Time: ___________
Analyzer Model No. _________________________ Serial No. _______________

		Initial values		Final values		
Calibration Gas Level	Certified Calibration gas value (indicate units)	System Response (indicate units)	System Bias[1] or Calibration Error[2] (% of calibration span)	System response (indicate units)	System Bias[1] or Calibration Error[2] (% of calibration span)	Drift (% of calibration span)
Low-level gas..........	....	....	....	....	....	
Upscale (high- or mid-) level gas..........	....	....	....	....	....	

[1] Refers to the pre- and post-run system bias checks of a non-dilution system.
[2] Refers to the pre- and post-run system calibration error checks of a dilution system.

TABLE 7E–3—EXAMPLE INTERFERENCE CHECK GAS CONCENTRATIONS

Potential interferent gas [1]	Concentrations [2] sample conditioning type	
	Hot wet	Dried
CO_2	5 and 15%	5 and 15%
H_2O	25%	1%
NO	15 ppmv	15 ppmv
NO_2	15 ppmv	15 ppmv
N_2O	10 ppmv	10 ppmv
CO	50 ppmv	50 ppmv
NH_3	10 ppmv	10 ppmv
CH_4	50 ppmv	50 ppmv
SO_2	20 ppmv	20 ppmv
H_2	50 ppmv	50 ppmv
HCl	10 ppmv	10 ppmv

[1] Any applicable gas may be eliminated or tested at a reduced level if the manufacturer has provided reliable means for limiting or scrubbing that gas to a specified level.

[2] As practicable, gas concentrations should be the highest expected at test sites.

TABLE 7E–4—INTERFERENCE RESPONSE

Date of Test: __________

Analyzer Type: __________

Model No.: __________

Serial No: __________

Calibration Span: __________

Test gas type	Concentration (ppm)	Analyzer response
	Sum of Responses	
	% of Calibration Span	

TABLE 7E–5—MANUFACTURER STABILITY TEST

Test description	Acceptance criteria (note 1)
Thermal Stability	Temperature range when drift does not exceed 3.0% of analyzer range over a 12-hour run when measured with NO_X present @ 80% of calibration span.
Fault Conditions	Identify conditions which, when they occur, result in performance which is not in compliance with the Manufacturer's Stability Test criteria. These are to be indicated visually or electrically to alert the operator of the problem.
Insensitivity to Supply Voltage Variations.	±10.0% (or manufacturers alternative) variation from nominal voltage must produce a drift of ≤2.0% of calibration span for either zero or concentration ≥80% NO_X present.
Analyzer Calibration Error.	For a low-, medium-, and high-calibration gas, the difference between the manufacturer certified value and the analyzer response in direct calibration mode, no more than 2.0% of calibration span.

Note 1: If the instrument is to be used as a Low Range analyzer, all tests must be performed at a calibration span of 20 ppm or less.

METHOD 8—DETERMINATION OF SULFURIC ACID AND SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5, and Method 6.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Sulfuric acid, including: Sulfuric acid (H_2SO_4) mist, Sulfur trioxide (SO_3).	7664–93–9, 7449–11–9	0.05 mg/m³ (0.03×10^{-7} lb/ft³).
Sulfur dioxide (SO_2)	7449–09–5	1.2 mg/m³ (3×10^{-9} lb/ft³).

1.2 Applicability. This method is applicable for the determination of H_2SO_4 (including H_2SO_4 mist and SO_3) and gaseous SO_2 emissions from stationary sources.

NOTE: Filterable particulate matter may be determined along with H_2SO_4 and SO_2 (subject to the approval of the Administrator) by inserting a heated glass fiber filter between the probe and isopropanol impinger (see section 6.1.1 of Method 6). If this option is chosen, particulate analysis is gravimetric only; sulfuric acid is not determined separately.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

A gas sample is extracted isokinetically from the stack. The H_2SO_4 and the SO_2 are separated, and both fractions are measured separately by the barium-thorin titration method.

3.0 Definitions [Reserved]

4.0 Interferences

4.1 Possible interfering agents of this method are fluorides, free ammonia, and dimethyl aniline. If any of these interfering agents is present (this can be determined by knowledge of the process), alternative methods, subject to the approval of the Administrator, are required.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive reagents. Same as Method 6, section 5.2.

6.0 Equipment and Supplies

6.1 Sample Collection. Same as Method 5, section 6.1, with the following additions and exceptions:

6.1.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 8–1; it is similar to the Method 5 sampling train, except that the filter position is different, and the filter holder does not have to be heated. See Method 5, section 6.1.1, for details and guidelines on operation and maintenance.

6.1.1.1 Probe Liner. Borosilicate or quartz glass, with a heating system to prevent visible condensation during sampling. Do not use metal probe liners.

6.1.1.2 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other gasket materials (*e.g.*, Teflon or Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The filter holder shall be placed between the first and second impingers. Do not heat the filter holder.

6.1.1.3 Impingers. Four, of the Greenburg-Smith design, as shown in Figure 8–1. The first and third impingers must have standard tips. The second and fourth impingers must be modified by replacing the insert with an approximately 13-mm (½-in.) ID glass tube, having an unconstricted tip located 13 mm (½ in.) from the bottom of the impinger. Similar collection systems, subject to the approval of the Administrator, may be used.

6.1.1.4 Temperature Sensor. Thermometer, or equivalent, to measure the temperature of the gas leaving the impinger train to within 1 °C (2 °F).

6.2 Sample Recovery. The following items are required for sample recovery:

6.2.1 Wash Bottles. Two polyethylene or glass bottles, 500-ml.

6.2.2 Graduated Cylinders. Two graduated cylinders (volumetric flasks may be used), 250-ml, 1-liter.

6.2.3 Storage Bottles. Leak-free polyethylene bottles, 1-liter size (two for each sampling run).

6.2.4 Trip Balance. 500-g capacity, to measure to ±0.5 g (necessary only if a moisture content analysis is to be done).

6.3 Analysis. The following items are required for sample analysis:

6.3.1 Pipettes. Volumetric 10-ml, 100-ml.

6.3.2 Burette. 50-ml.

6.3.3 Erlenmeyer Flask. 250-ml (one for each sample, blank, and standard).

6.3.4 Graduated Cylinder. 100-ml.

6.3.5 Dropping Bottle. To add indicator solution, 125-ml size.

7.0 Reagents and Standards

NOTE: Unless otherwise indicated, all reagents are to conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Otherwise, use the best available grade.

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Filters and Silica Gel. Same as in Method 5, sections 7.1.1 and 7.1.2, respectively.

7.1.2 Water. Same as in Method 6, section 7.1.1.

7.1.3 Isopropanol, 80 Percent by Volume. Mix 800 ml of isopropanol with 200 ml of water.

NOTE: Check for peroxide impurities using the procedure outlined in Method 6, section 7.1.2.1.

7.1.4 Hydrogen Peroxide (H^2O^2), 3 Percent by Volume. Dilute 100 ml of 30 percent H_2O_2) to 1 liter with water. Prepare fresh daily.

7.1.5 Crushed Ice.

7.2 Sample Recovery. The reagents and standards required for sample recovery are:

7.2.1 Water. Same as in section 7.1.2.

7.2.2 Isopropanol, 80 Percent. Same as in section 7.1.3.

7.3 Sample Analysis. Same as Method 6, section 7.3.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Preparation. Same as Method 5, section 8.1, except that filters should be inspected but need not be desiccated, weighed, or identified. If the effluent gas can be considered dry (i.e., moisture-free), the silica gel need not be weighed.

8.2 Preliminary Determinations. Same as Method 5, section 8.2.

8.3 Preparation of Sampling Train. Same as Method 5, section 8.3, with the following exceptions:

8.3.1 Use Figure 8–1 instead of Figure 5–1.

8.3.2 Replace the second sentence of Method 5, section 8.3.1 with: Place 100 ml of 80 percent isopropanol in the first impinger, 100 ml of 3 percent H_2O_2 in both the second and third impingers; retain a portion of each reagent for use as a blank solution. Place about 200 g of silica gel in the fourth impinger.

8.3.3 Ignore any other statements in section 8.3 of Method 5 that are obviously not applicable to the performance of Method 8.

NOTE: If moisture content is to be determined by impinger analysis, weigh each of the first three impingers (plus absorbing solution) to the nearest 0.5 g, and record these weights. Weigh also the silica gel (or silica gel plus container) to the nearest 0.5 g, and record.)

8.4 Metering System Leak-Check Procedure. Same as Method 5, section 8.4.1.

8.5 Pretest Leak-Check Procedure. Follow the basic procedure in Method 5, section 8.4.2, noting that the probe heater shall be adjusted to the minimum temperature required to prevent condensation, and also that verbage such as "* * * plugging the inlet to the filter holder * * * " found in section 8.4.2.2 of Method 5 shall be replaced by " * * * plugging the inlet to the first impinger * * * ". The pretest leak-check is recommended, but is not required.

8.6 Sampling Train Operation. Follow the basic procedures in Method 5, section 8.5, in conjunction with the following special instructions:

8.6.1 Record the data on a sheet similar to that shown in Figure 8–2 (alternatively, Figure 5–2 in Method 5 may be used). The sampling rate shall not exceed 0.030 m^3/min (1.0 cfm) during the run. Periodically during the test, observe the connecting line between the probe and first impinger for signs of condensation. If condensation does occur, adjust the probe heater setting upward to the minimum temperature required to prevent condensation. If component changes become necessary during a run, a leak-check shall be performed immediately before each change, according to the procedure outlined in section 8.4.3 of Method 5 (with appropriate modifications, as mentioned in section 8.5 of this method); record all leak rates. If the leakage rate(s) exceeds the specified rate, the tester shall either void the run or plan to correct the sample volume as outlined in section 12.3 of Method 5. Leak-checks immediately after component changes are recommended, but not required. If these leak-checks are performed, the procedure in section 8.4.2 of Method 5 (with appropriate modifications) shall be used.

8.6.2 After turning off the pump and recording the final readings at the conclusion of each run, remove the probe from the stack. Conduct a post-test (mandatory) leak-check as outlined in section 8.4.4 of Method 5 (with appropriate modifications), and record the leak rate. If the post-test leakage rate exceeds the specified acceptable rate, either correct the sample volume, as outlined in section 12.3 of Method 5, or void the run.

8.6.3 Drain the ice bath and, with the probe disconnected, purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the average flow rate used for sampling.

NOTE: Clean ambient air can be provided by passing air through a charcoal filter. Alternatively, ambient air (without cleaning) may be used.

8.7 Calculation of Percent Isokinetic. Same as Method 5, section 8.6.

8.8 Sample Recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. Treat the samples as follows:

8.8.1 Container No. 1.

8.8.1.1 If a moisture content analysis is to be performed, clean and weigh the first impinger (plus contents) to the nearest 0.5 g, and record this weight.

8.8.1.2 Transfer the contents of the first impinger to a 250-ml graduated cylinder. Rinse the probe, first impinger, all connecting glassware before the filter, and the front half of the filter holder with 80 percent isopropanol. Add the isopropanol rinse solution to the cylinder. Dilute the contents of the cylinder to 225 ml with 80 percent isopropanol, and transfer the cylinder contents to the storage container. Rinse the cylinder with 25 ml of 80 percent isopropanol, and transfer the rinse to the storage container. Add the filter to the solution in the storage container and mix. Seal the container to protect the solution against evaporation. Mark the level of liquid on the container, and identify the sample container.

8.8.2 Container No. 2.

8.8.2.1 If a moisture content analysis is to be performed, clean and weigh the second and third impingers (plus contents) to the nearest 0.5 g, and record the weights. Also, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g, and record the weight.

8.8.2.2 Transfer the solutions from the second and third impingers to a 1-liter graduated cylinder. Rinse all connecting glassware (including back half of filter holder) between the filter and silica gel impinger with water, and add this rinse water to the cylinder. Dilute the contents of the cylinder to 950 ml with water. Transfer the solution to a storage container. Rinse the cylinder with 50 ml of water, and transfer the rinse to the storage container. Mark the level of liquid on the container. Seal and identify the sample container.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
7.1.3	Isopropanol check	Ensure acceptable level of peroxide impurities in isopropanol.
8.4, 8.5, 10.1	Sampling equipment leak-check and calibration.	Ensure accurate measurement of stack gas flow rate, sample volume.
10.2	Barium standard solution standardization	Ensure normality determination.
11.2	Replicate titrations	Ensure precision of titration determinations.

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

10.1 Sampling Equipment. Same as Method 5, section 10.0.

10.2 Barium Standard Solution. Same as Method 6, section 10.5.

11.0 Analytical Procedure

11.1. Sample Loss. Same as Method 6, section 11.1.

11.2. Sample Analysis.

11.2.1 Container No. 1. Shake the container holding the isopropanol solution and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample aliquot. Pipette a 100-ml aliquot of this solution into a 250-ml Erlenmeyer flask, add 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium standard solution. Repeat the titration with a second aliquot of sample, and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

11.2.2 Container No. 2. Thoroughly mix the solution in the container holding the contents of the second and third impingers. Pipette a 10-ml aliquot of sample into a 250-ml Erlenmeyer flask. Add 40 ml of isopropanol, 2 to 4 drops of thorin indicator, and titrate to a pink endpoint using 0.0100 N barium standard solution. Repeat the titration with a second aliquot of sample, and average the titration values. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is greater.

11.2.3 Blanks. Prepare blanks by adding 2 to 4 drops of thorin indicator to 100 ml of 80 percent isopropanol. Titrate the blanks in the same manner as the samples.

12.0 Data Analysis and Calculations

Carry out calculations retaining at least one extra significant figure beyond that of the acquired data. Round off figures after final calculation.

12.1 Nomenclature. Same as Method 5, section 12.1, with the following additions and exceptions:

$C_{H_2SO_4}$ = Sulfuric acid (including SO_3) concentration, g/dscm (lb/dscf).

C_{SO_2} = Sulfur dioxide concentration, g/dscm (lb/dscf).

N = Normality of barium perchlorate titrant, meq/ml.

V_a = Volume of sample aliquot titrated, 100 ml for H_2SO_4 and 10 ml for SO_2.

V_{soln} = Total volume of solution in which the sample is contained, 1000 ml for the SO_2 sample and 250 ml for the H_2SO_4 sample.

V_t = Volume of barium standard solution titrant used for the sample, ml.

V_{tb} = Volume of barium standard solution titrant used for the blank, ml.

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 8–2).

12.3 Dry Gas Volume. Same as Method 5, section 12.3.

12.4 Volume of Water Vapor Condensed and Moisture Content. Calculate the volume of water vapor using Equation 5–2 of Method 5; the weight of water collected in the impingers and silica gel can be converted directly to milliliters (the specific gravity of water is 1 g/ml). Calculate the moisture content of the stack gas (B_{ws}) using Equation 5–3 of Method 5. The note in section 12.5 of Method 5 also applies to this method. Note that if the effluent gas stream can be considered dry, the volume of water vapor and moisture content need not be calculated.

12.5 Sulfuric Acid Mist (Including SO_3) Concentration.

$$C_{H_2SO_4} = K_3\left[N\left(V_t - V_{tb}\right)\left(V_{soln}/V_a\right)\right]/V_{m(std)} \qquad \text{Eq. 8-1}$$

Where:

K_3 = 0.04904 g/meq for metric units,

$K_3 = 1.081 \times 10^{-4}$ lb/meq for English units.

12.6 Sulfur Dioxide Concentration.

$$C_{SO_2} = K_4\left[N\left(V_t - V_{tb}\right)\left(V_{soln}/V_a\right)\right]/V_{m(std)} \qquad \text{Eq. 8-2}$$

Where:

K_4 = 0.03203 g/meq for metric units,

$K_4 = 7.061 \times 10^{-5}$ lb/meq for English units.

12.7 Isokinetic Variation. Same as Method 5, section 12.11.

12.8 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in sections 12.6 and 12.7 of Method 2.

13.0 Method Performance

13.1 Analytical Range. Collaborative tests have shown that the minimum detectable limits of the method are 0.06 mg/m³ (4×10^{-9} lb/ft³) for H_2SO_4 and 1.2 mg/m³ (74×10^{-9} lb/ft³) for SO_2. No upper limits have been established. Based on theoretical calculations for 200 ml of 3 percent H_2O_2 solution, the upper concentration limit for SO_2 in a 1.0 m³ (35.3 ft³) gas sample is about 12,000 mg/m³ (7.7×10^{-4} lb/ft³). The upper limit can be extended by increasing the quantity of peroxide solution in the impingers.

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as section 17.0 of Methods 5 and 6.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

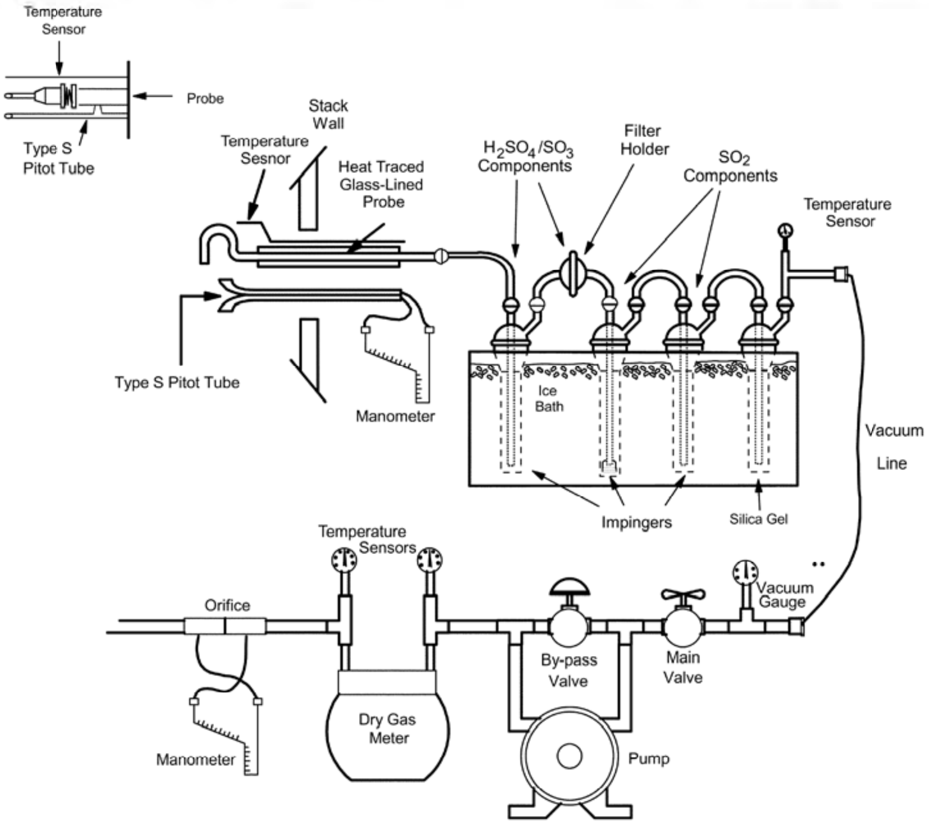


Figure 8-1. Sulfuric Acid Sampling Train

Plant __________
Location __________
Operator __________
Date __________
Run No. __________
Sample box No. __________
Meter box No. __________
Meter ΔH@ __________
C factor __________
Pitot tube coefficient, C_p __________

SCHEMATIC OF STACK CROSS SECTION

Ambient temperature __________
Barometric pressure __________
Assumed moisture, % __________
Probe length, (ft.) __________
Nozzle identification No. __________
Average calibrated nozzle diameter, (in.) __________
Probe heater setting __________
Leak rate, (cfm) __________
Static pressure, (in. Hg) __________
Probe liner material __________
Filter No. __________

Traverse point number	Sampling time	Vacuum	Stack temperature	Velocity head	Pressure differential across orifice meter	Gas meter reading	Gas sample temperature at dry gas meter		Filter temperature	Temperature of gas leaving condenser or last impinger
							Inlet	Outlet		
	min.	(in.Hg)	(T_s) (°F)	(ΔP_s) (in. H_2O)	(in. H_2O)	(ft³)	(°F)	(°F)	(°F)	(°F)
Total							Avg.	Avg.		
Average							Avg.			

Figure 8-2. Field Data Sheet.

METHOD 9—VISUAL DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES

Many stationary sources discharge visible emissions into the atmosphere; these emissions are usually in the shape of a plume. This method involves the determination of plume opacity by qualified observers. The method includes procedures for the training and certification of observers, and procedures to be used in the field for determination of plume opacity. The appearance of a plume as viewed by an observer depends upon a number of variables, some of which may be controllable and some of which may not be controllable in the field. Variables which can be controlled to an extent to which they no longer exert a significant influence upon plume appearance include: Angle of the observer with respect to the plume; angle of the observer with respect to the sun; point of observation of attached and detached steam plume; and angle of the observer with respect to a plume emitted from a rectangular stack with a large length to width ratio. The method includes specific criteria applicable to these variables.

Other variables which may not be controllable in the field are luminescence and color contrast between the plume and the background against which the plume is viewed. These variables exert an influence upon the appearance of a plume as viewed by an observer, and can affect the ability of the observer to accurately assign opacity values to the observed plume. Studies of the theory of plume opacity and field studies have demonstrated that a plume is most visible and presents the greatest apparent opacity when viewed against a contrasting background. It follows from this, and is confirmed by field trials, that the opacity of a plume, viewed under conditions where a contrasting background is present can be assigned with the greatest degree of accuracy. However, the potential for a positive error is also the greatest when a plume is viewed under such contrasting conditions. Under conditions presenting a less contrasting background, the apparent opacity of a plume is less and approaches zero as the color and luminescence contrast decrease toward zero. As a result, significant negative bias and negative errors can be made when a plume is viewed under less contrasting conditions. A negative bias decreases rather than increases the possibility that a plant operator will be cited for a violation of opacity standards due to observer error.

Studies have been undertaken to determine the magnitude of positive errors which can be made by qualified observers while reading plumes under contrasting conditions and using the procedures set forth in this method. The results of these studies (field trials) which involve a total of 769 sets of 25 readings each are as follows:

(1) For black plumes (133 sets at a smoke generator), 100 percent of the sets were read with a positive error[1] of less than 7.5 percent opacity; 99 percent were read with a positive error of less than 5 percent opacity.

(2) For white plumes (170 sets at a smoke generator, 168 sets at a coal-fired power plant, 298 sets at a sulfuric acid plant), 99 percent of the sets were read with a positive error of less than 7.5 percent opacity; 95 percent were read with a positive error of less than 5 percent opacity.

The positive observational error associated with an average of twenty-five readings is therefore established. The accuracy of the method must be taken into account when determining possible violations of applicable opacity standards.

1. Principle and Applicability

1.1 Principle. The opacity of emissions from stationary sources is determined visually by a qualified observer.

1.2 Applicability. This method is applicable for the determination of the opacity of emissions from stationary sources pursuant to §60.11(b) and for qualifying observers for visually determining opacity of emissions.

2. Procedures

The observer qualified in accordance with section 3 of this method shall use the following procedures for visually determining the opacity of emissions:

2.1 Position. The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140° sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction, and when observing opacity of emissions from rectangular outlets (e.g., roof monitors, open baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g., stub stacks on baghouses).

2.2 Field Records. The observer shall record the name of the plant, emission location, type facility, observer's name and affiliation, a sketch of the observer's position relative to the source, and the date on a field data sheet (Figure 9–1). The time, estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

2.3 Observations. Opacity observations shall be made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

2.3.1 Attached Steam Plumes. When condensed water vapor is present within the

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[1] For a set, positive error = average opacity determined by observers' 25 observations—average opacity determined from transmissometer's 25 recordings.

plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible. The observer shall record the approximate distance from the emission outlet to the point in the plume at which the observations are made.

2.3.2 Detached Steam Plume. When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

2.4 Recording Observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on an observational record sheet. (See Figure 9–2 for an example.) A minimum of 24 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

2.5 Data Reduction. Opacity shall be determined as an average of 24 consecutive observations recorded at 15-second intervals. Divide the observations recorded on the record sheet into sets of 24 consecutive observations. A set is composed of any 24 consecutive observations. Sets need not be consecutive in time and in no case shall two sets overlap. For each set of 24 observations, calculate the average by summing the opacity of the 24 observations and dividing this sum by 24. If an applicable standard specifies an averaging time requiring more than 24 observations, calculate the average for all observations made during the specified time period. Record the average opacity on a record sheet. (See Figure 9–1 for an example.)

3. Qualifications and Testing

3.1 Certification Requirements. To receive certification as a qualified observer, a candidate must be tested and demonstrate the ability to assign opacity readings in 5 percent increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent opacity on any one reading and an average error not to exceed 7.5 percent opacity in each category. Candidates shall be tested according to the procedures described in section 3.2. Smoke generators used pursuant to section 3.2 shall be equipped with a smoke meter which meets the requirements of section 3.3.

The certification shall be valid for a period of 6 months, at which time the qualification procedure must be repeated by any observer in order to retain certification.

3.2 Certification Procedure. The certification test consists of showing the candidate a complete run of 50 plumes—25 black plumes and 25 white plumes—generated by a smoke generator. Plumes within each set of 25 black and 25 white runs shall be presented in random order. The candidate assigns an opacity value to each plume and records his observation on a suitable form. At the completion of each run of 50 readings, the score of the candidate is determined. If a candidate fails to qualify, the complete run of 50 readings must be repeated in any retest. The smoke test may be administered as part of a smoke school or training program, and may be preceded by training or familiarization runs of the smoke generator during which candidates are shown black and white plumes of known opacity.

3.3 Smoke Generator Specifications. Any smoke generator used for the purposes of section 3.2 shall be equipped with a smoke meter installed to measure opacity across the diameter of the smoke generator stack. The smoke meter output shall display instack opacity based upon a pathlength equal to the stack exit diameter, on a full 0 to 100 percent chart recorder scale. The smoke meter optical design and performance shall meet the specifications shown in Table 9–1. The smoke meter shall be calibrated as prescribed in section 3.3.1 prior to the conduct of each smoke reading test. At the completion of each test, the zero and span drift shall be checked and if the drift exceeds ±1 percent opacity, the condition shall be corrected prior to conducting any subsequent test runs. The smoke meter shall be demonstrated, at the time of installation, to meet the specifications listed in Table 9–1. This demonstration shall be repeated following any subsequent repair or replacement of the photocell or associated electronic circuitry including the chart recorder or output meter, or every 6 months, whichever occurs first.

TABLE 9–1—SMOKE METER DESIGN AND PERFORMANCE SPECIFICATIONS

Parameter	Specification
a. Light source	Incandescent lamp operated at nominal rated voltage.
b. Spectral response of photocell.	Photopic (daylight spectral response of the human eye—Citation 3).
c. Angle of view	15° maximum total angle.
d. Angle of projection	15° maximum total angle.
e. Calibration error	±3% opacity, maximum.
f. Zero and span drift	±1% opacity, 30 minutes.
g. Response time	5 seconds.

3.3.1 Calibration. The smoke meter is calibrated after allowing a minimum of 30 minutes warmup by alternately producing simulated opacity of 0 percent and 100 percent. When stable response at 0 percent or 100 percent is noted, the smoke meter is adjusted to produce an output of 0 percent or 100 percent, as appropriate. This calibration shall be repeated until stable 0 percent and 100 percent readings are produced without adjustment. Simulated 0 percent and 100 percent opacity values may be produced by alternately switching the power to the light source on and off while the smoke generator is not producing smoke.

3.3.2 Smoke Meter Evaluation. The smoke meter design and performance are to be evaluated as follows:

3.3.2.1 Light Source. Verify from manufacturer's data and from voltage measurements made at the lamp, as installed, that the lamp is operated within ±5 percent of the nominal rated voltage.

3.3.2.2 Spectral Response of Photocell. Verify from manufacturer's data that the photocell has a photopic response; i.e., the spectral sensitivity of the cell shall closely approximate the standard spectral-luminosity curve for photopic vision which is referenced in (b) of Table 9–1.

FIGURE 9-1
RECORD OF VISUAL DETERMINATION OF OPACITY

PAGE____of____

COMPANY__________
LOCATION__________
TEST NUMBER__________
DATE__________
TYPE FACILITY__________
CONTROL DEVICE__________

HOURS OF OBSERVATION__________
OBSERVER__________
OBSERVER CERTIFICATION DATE__________
OBSERVER AFFILIATION__________
POINT OF EMISSIONS__________
HEIGHT OF DISCHARGE POINT__________

	Initial			Final
CLOCK TIME				
OBSERVER LOCATION Distance to Discharge				
Direction from Discharge				
Height of Observation Point				
BACKGROUND DESCRIPTION				
WEATHER CONDITIONS Wind Direction				
Wind Speed				
Ambient Temperature				
SKY CONDITIONS (clear, overcast, % clouds, etc.)				
PLUME DESCRIPTION Color				
Distance Visible				
OTHER INFORMATION				

SUMMARY OF AVERAGE OPACITY

Set Number	Time	Opacity	
	Start--End	Sum	Average

Readings ranged from ____ to ____ % opacity

The source was/was not in compliance with ____ at the time evaluation was made.

FIGURE 9-2—OBSERVATION RECORD

Page ____ of ____

Company ....
Location ....
Test Number ....
Date.

Observer ....
Type facility ....
Point of emissions ....

Hr.	Min.	Seconds				Steam plume (check if applicable)		Comments
		0	15	30	45	Attached	Detached	
	0							
	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
	9							
	10							
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	22							
	23							
	24							
	25							
	26							
	27							
	28							
	29							

FIGURE 9–2—OBSERVATION RECORD (CONTINUED)

Page ____ of ____

Company ....
Location ....
Test Number ....
Date.

Observer ....
Type facility ....
Point of emissions ....

Hr.	Min.	Seconds				Steam plume (check if applicable)		Comments
		0	15	30	45	Attached	Detached	
	30							

Hr.	Min.	Seconds				Steam plume (check if applicable)		Comments
		0	15	30	45	Attached	Detached	
	31							
	32							
	33							
	34							
	35							
	36							
	37							
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3.3.2.3 Angle of View. Check construction geometry to ensure that the total angle of view of the smoke plume, as seen by the photocell, does not exceed 15°. The total angle of view may be calculated from: $\theta = 2 \tan^{-1} d/2L$, where θ = total angle of view; d = the sum of the photocell diameter + the diameter of the limiting aperture; and L = the distance from the photocell to the limiting aperture. The limiting aperture is the point in the path between the photocell and the smoke plume where the angle of view is most restricted. In smoke generator smoke meters this is normally an orifice plate.

3.3.2.4 Angle of Projection. Check construction geometry to ensure that the total angle of projection of the lamp on the smoke plume does not exceed 15°. The total angle of projection may be calculated from: $\theta = 2 \tan^{-1} d/2L$, where θ = total angle of projection; d = the sum of the length of the lamp filament + the diameter of the limiting aperture; and L = the distance from the lamp to the limiting aperture.

3.3.2.5 Calibration Error. Using neutral-density filters of known opacity, check the error between the actual response and the theoretical linear response of the smoke meter. This check is accomplished by first calibrating the smoke meter according to 3.3.1 and then inserting a series of three neutral-density filters of nominal opacity of 20, 50, and 75 percent in the smoke meter pathlength. Filters calibrated within ±2 percent shall be used. Care should be taken when inserting the filters to prevent stray light from affecting the meter. Make a total of five nonconsecutive readings for each filter. The maximum error on any one reading shall be 3 percent opacity.

3.3.2.6 Zero and Span Drift. Determine the zero and span drift by calibrating and operating the smoke generator in a normal manner over a 1-hour period. The drift is measured by checking the zero and span at the end of this period.

3.3.2.7 Response Time. Determine the response time by producing the series of five simulated 0 percent and 100 percent opacity values and observing the time required to reach stable response. Opacity values of 0 percent and 100 percent may be simulated by alternately switching the power to the light source off and on while the smoke generator is not operating.

4. Bibliography

1. Air Pollution Control District Rules and Regulations, Los Angeles County Air Pollution Control District, Regulation IV, Prohibitions, Rule 50.

2. Weisburd, Melvin I., Field Operations and Enforcement Manual for Air, U.S. Environmental Protection Agency, Research Triangle Park, NC. APTD–1100, August 1972, pp. 4.1–4.36.

3. Condon, E.U., and Odishaw, H., Handbook of Physics, McGraw-Hill Co., New York, NY, 1958, Table 3.1, p. 6–52.

ALTERNATE METHOD 1—DETERMINATION OF THE OPACITY OF EMISSIONS FROM STATIONARY SOURCES REMOTELY BY LIDAR

This alternate method provides the quantitative determination of the opacity of an emissions plume remotely by a mobile lidar system (laser radar; Light Detection and Ranging). The method includes procedures for the calibration of the lidar and procedures to be used in the field for the lidar determination of plume opacity. The lidar is used to measure plume opacity during either day or nighttime hours because it contains its own pulsed light source or transmitter. The operation of the lidar is not dependent upon ambient lighting conditions (light, dark, sunny or cloudy).

The lidar mechanism or technique is applicable to measuring plume opacity at numerous wavelengths of laser radiation. However, the performance evaluation and calibration test results given in support of this method apply only to a lidar that employs a ruby (red light) laser [Reference 5.1].

1. Principle and Applicability

1.1 Principle. The opacity of visible emissions from stationary sources (stacks, roof vents, etc.) is measured remotely by a mobile lidar (laser radar).

1.2 Applicability. This method is applicable for the remote measurement of the opacity of visible emissions from stationary sources during both nighttime and daylight conditions, pursuant to 40 CFR §60.11(b). It is also applicable for the calibration and performance verification of the mobile lidar for the measurement of the opacity of emissions. A performance/design specification for a basic lidar system is also incorporated into this method.

1.3 Definitions.

Azimuth angle: The angle in the horizontal plane that designates where the laser beam is pointed. It is measured from an arbitrary fixed reference line in that plane.

Backscatter: The scattering of laser light in a direction opposite to that of the incident laser beam due to reflection from particulates along the beam's atmospheric path which may include a smoke plume.

Backscatter signal: The general term for the lidar return signal which results from laser light being backscattered by atmospheric and smoke plume particulates.

Convergence distance: The distance from the lidar to the point of overlap of the lidar receiver's field-of-view and the laser beam.

Elevation angle: The angle of inclination of the laser beam referenced to the horizontal plane.

Far region: The region of the atmosphere's path along the lidar line-of-sight beyond or behind the plume being measured.

Lidar: Acronym for Light Detection and Ranging.

Lidar range: The range or distance from the lidar to a point of interest along the lidar line-of-sight.

Near region: The region of the atmospheric path along the lidar line-of-sight between the lidar's convergence distance and the plume being measured.

Opacity: One minus the optical transmittance of a smoke plume, screen target, etc.

Pick interval: The time or range intervals in the lidar backscatter signal whose minimum average amplitude is used to calculate opacity. Two pick intervals are required, one in the near region and one in the far region.

Plume: The plume being measured by lidar.

Plume signal: The backscatter signal resulting from the laser light pulse passing through a plume.

$1/R^2$Correction: The correction made for the systematic decrease in lidar backscatter signal amplitude with range.

Reference signal: The backscatter signal resulting from the laser light pulse passing through ambient air.

Sample interval: The time period between successive samples for a digital signal or between successive measurements for an analog signal.

Signal spike: An abrupt, momentary increase and decrease in signal amplitude.

Source: The source being tested by lidar.

Time reference: The time (t_o) when the laser pulse emerges from the laser, used as the reference in all lidar time or range measurements.

2. Procedures

The mobile lidar calibrated in accordance with Paragraph 3 of this method shall use the following procedures for remotely measuring the opacity of stationary source emissions:

2.1 Lidar Position. The lidar shall be positioned at a distance from the plume sufficient to provide an unobstructed view of the source emissions. The plume must be at a range of at least 50 meters or three consecutive pick intervals (whichever is greater) from the lidar's transmitter/receiver convergence distance along the line-of-sight. The maximum effective opacity measurement distance of the lidar is a function of local atmospheric conditions, laser beam diameter, and plume diameter. The test position of the lidar shall be selected so that the diameter of the laser beam at the measurement point within the plume shall be no larger than three-fourths the plume diameter. The beam diameter is calculated by Equation (AM1–1):

$$D(\text{lidar}) = A + R\varphi \leq 0.75\ D(\text{Plume}) \quad \text{(AM1–1)}$$

Where:

D(Plume) = diameter of the plume (cm),

φ = laser beam divergence measured in radians

R = range from the lidar to the source (cm)

D(Lidar) = diameter of the laser beam at range R (cm),

A = diameter of the laser beam or pulse where it leaves the laser.

The lidar range, R, is obtained by aiming and firing the laser at the emissions source structure immediately below the outlet. The range value is then determined from the backscatter signal which consists of a signal spike (return from source structure) and the atmospheric backscatter signal [Reference 5.1]. This backscatter signal should be recorded.

When there is more than one source of emissions in the immediate vicinity of the plume, the lidar shall be positioned so that the laser beam passes through only a single plume, free from any interference of the other plumes for a minimum of 50 meters or three consecutive pick intervals (whichever is greater) in each region before and beyond the plume along the line-of-sight (determined from the backscatter signals). The lidar shall initially be positioned so that its line-of-sight is approximately perpendicular to the plume.

When measuring the opacity of emissions from rectangular outlets (e.g., roof monitors, open baghouses, noncircular stacks, etc.), the lidar shall be placed in a position so that its line-of-sight is approximately perpendicular to the longer (major) axis of the outlet.

2.2 Lidar Operational Restrictions. The lidar receiver shall not be aimed within an angle of ±15° (cone angle) of the sun.

This method shall not be used to make opacity measurements if thunderstorms, snowstorms, hail storms, high wind, high-ambient dust levels, fog or other atmospheric conditions cause the reference signals to consistently exceed the limits specified in section 2.3.

2.3 Reference Signal Requirements. Once placed in its proper position for opacity measurement, the laser is aimed and fired with the line-of-sight near the outlet height and rotated horizontally to a position clear of the source structure and the associated plume. The backscatter signal obtained from this position is called the ambient-air or reference signal. The lidar operator shall inspect this signal [Section V of Reference 5.1] to: (1) determine if the lidar line-of-sight is free from interference from other plumes and from physical obstructions such as cables, power lines, etc., for a minimum of 50 meters or three consecutive pick intervals (whichever is greater) in each region before and beyond the plume, and (2) obtain a qualitative measure of the homogeneity of the ambient air by noting any signal spikes.

Should there be any signal spikes on the reference signal within a minimum of 50 meters or three consecutive pick intervals (whichever is greater) in each region before and beyond the plume, the laser shall be fired three more times and the operator shall inspect the reference signals on the display. If the spike(s) remains, the azimuth angle shall be changed and the above procedures conducted again. If the spike(s) disappears in all three reference signals, the lidar line-of-sight is acceptable if there is shot-to-shot consistency and there is no interference from other plumes.

Shot-to-shot consistency of a series of reference signals over a period of twenty seconds is verified in either of two ways. (1) The lidar operator shall observe the reference signal amplitudes. For shot-to-shot consistency the ratio of R_f to R_n [amplitudes of the near and far region pick intervals (Section 2.6.1)] shall vary by not more than ±6% between shots; or (2) the lidar operator shall accept any one of the reference signals and treat the other two as plume signals; then the opacity for each of the subsequent reference signals is calculated (Equation AM1–2). For shot-to-shot consistency, the opacity values shall be within ±3% of 0% opacity and the associated S_o values less than or equal to 8% (full scale) [Section 2.6].

If a set of reference signals fails to meet the requirements of this section, then all plume signals [Section 2.4] from the last set of acceptable reference signals to the failed set shall be discarded.

2.3.1 Initial and Final Reference Signals. Three reference signals shall be obtained within a 90-second time period prior to any data run. A final set of three reference signals shall be obtained within three (3) minutes after the completion of the same data run.

2.3.2 Temporal Criterion for Additional Reference Signals. An additional set of reference signals shall be obtained during a data run if there is a change in wind direction or plume drift of 30° or more from the direction that was prevalent when the last set of reference signals was obtained. An additional set of reference signals shall also be obtained if there is an increase in value of S_{In} (near region standard deviation, Equation AM1–5) or S_{If} (far region standard deviation, Equation AM1–6) that is greater than 6% (full scale) over the respective values calculated from the immediately previous plume signal, and this increase in value remains for 30 seconds or longer. An additional set of reference signals shall also be obtained if there is a change in amplitude in either the near or the far region of the plume signal, that is greater than 6% of the near signal amplitude and this change in amplitude remains for 30 seconds or more.

2.4 Plume Signal Requirements. Once properly aimed, the lidar is placed in operation with the nominal pulse or firing rate of six pulses/minute (1 pulse/10 seconds). The lidar operator shall observe the plume backscatter signals to determine the need for additional reference signals as required by section 2.3.2. The plume signals are recorded from lidar start to stop and are called a data run. The length of a data run is determined by operator discretion. Short-term stops of the lidar to record additional reference signals do not constitute the end of a data run if plume signals are resumed within 90 seconds after the reference signals have been recorded, and the total stop or interrupt time does not exceed 3 minutes.

2.4.1 Non-hydrated Plumes. The laser shall be aimed at the region of the plume which displays the greatest opacity. The lidar operator must visually verify that the laser is aimed clearly above the source exit structure.

2.4.2 Hydrated Plumes. The lidar will be used to measure the opacity of hydrated or so-called steam plumes. As listed in the reference method, there are two types, i.e., attached and detached steam plumes.

2.4.2.1 Attached Steam Plumes. When condensed water vapor is present within a plume, lidar opacity measurements shall be made at a point within the residual plume where the condensed water vapor is no longer visible. The laser shall be aimed into the most dense region (region of highest opacity) of the residual plume.

During daylight hours the lidar operator locates the most dense portion of the residual plume visually. During nighttime hours a high-intensity spotlight, night vision scope, or low light level TV, etc., can be used as an aid to locate the residual plume. If visual determination is ineffective, the lidar may be used to locate the most dense region of the residual plume by repeatedly measuring opacity, along the longitudinal axis or center of the plume from the emissions outlet to a point just beyond the steam plume. The lidar operator should also observe color differences and plume reflectivity to ensure that the lidar is aimed completely within the residual plume. If the operator does not obtain a clear indication of the location of the residual plume, this method shall not be used.

Once the region of highest opacity of the residual plume has been located, aiming adjustments shall be made to the laser line-of-sight to correct for the following: movement to the region of highest opacity out of the lidar line-of-sight (away from the laser beam) for more than 15 seconds, expansion of the steam plume (air temperature lowers and/or relative humidity increases) so that it just begins to encroach on the field-of-view of the lidar's optical telescope receiver, or a decrease in the size of the steam plume (air temperature higher and/or relative humidity decreases) so that regions within the residual plume whose opacity is higher than the one being monitored, are present.

2.4.2.2 Detached Steam Plumes. When the water vapor in a hydrated plume condenses and becomes visible at a finite distance from the stack or source emissions outlet, the opacity of the emissions shall be measured in the region of the plume clearly above the emissions outlet and below condensation of the water vapor.

During daylight hours the lidar operators can visually determine if the steam plume is detached from the stack outlet. During nighttime hours a high-intensity spotlight, night vision scope, low light level TV, etc., can be used as an aid in determining if the steam plume is detached. If visual determination is ineffective, the lidar may be used to determine if the steam plume is detached by repeatedly measuring plume opacity from the outlet to the steam plume along the plume's longitudinal axis or center line. The lidar operator should also observe color differences and plume reflectivity to detect a detached plume. If the operator does not obtain a clear indication of the location of the detached plume, this method shall not be used to make opacity measurements between the outlet and the detached plume.

Once the determination of a detached steam plume has been confirmed, the laser shall be aimed into the region of highest opacity in the plume between the outlet and the formation of the steam plume. Aiming adjustments shall be made to the lidar's line-of-sight within the plume to correct for changes in the location of the most dense region of the plume due to changes in wind direction and speed or if the detached steam plume moves closer to the source outlet encroaching on the most dense region of the plume. If the detached steam plume should move too close to the source outlet for the lidar to make interference-free opacity measurements, this method shall not be used.

2.5 Field Records. In addition to the recording recommendations listed in other sections of this method the following records should be maintained. Each plume measured should be uniquely identified. The name of the facility, type of facility, emission source type, geographic location of the lidar with respect to the plume, and plume characteristics should be recorded. The date of the test, the time period that a source was monitored, the time (to the nearest second) of each opacity measurement, and the sample interval should also be recorded. The wind speed, wind direction, air temperature, relative humidity, visibility (measured at the lidar's position), and cloud cover should be recorded at the beginning and end of each time period for a given source. A small sketch depicting the location of the laser beam within the plume should be recorded.

If a detached or attached steam plume is present at the emissions source, this fact should be recorded. Figures AM1–I and AM1–II are examples of logbook forms that may be used to record this type of data. Magnetic tape or paper tape may also be used to record data.

LIDAR LOG CONTROL NUMBER TABULATION

Log Book Number-________

(Assign a CONTROL NUMBER to each individual source under test)

CONTROL NUMBER	DATE ASSIGNED	PROJECT	CITY, STATE

continued on next page

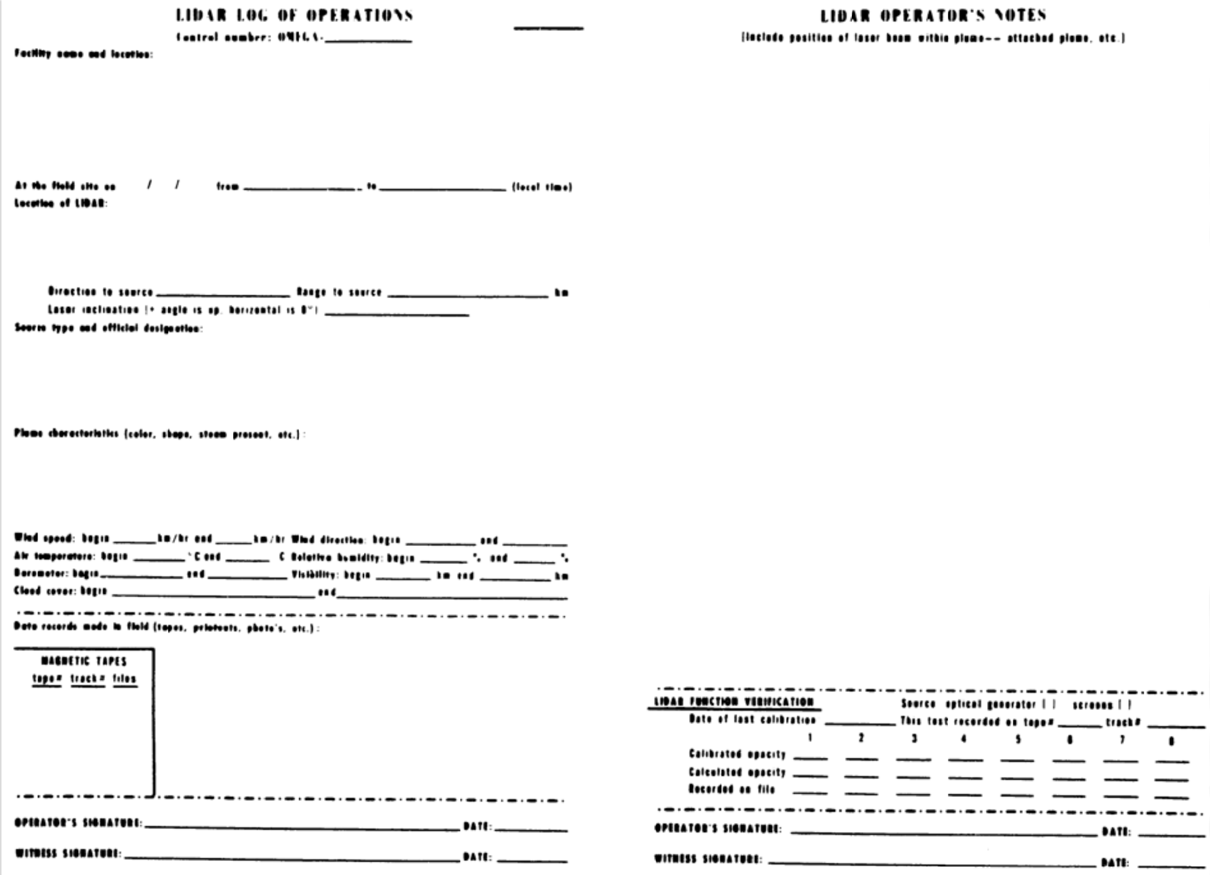
LIDAR LOG CONTROL NUMBER TABULATION (cont')

(Assign a CONTROL NUMBER to each individual source under test)

CONTROL NUMBER	DATE ASSIGNED	PROJECT	CITY, STATE

Next Log Book Number-________

Figure AM1-I Lidar Log Control Number Tabulation



LIDAR LOG OF OPERATIONS

Control number: OMEGA-__________

Facility name and location:

At the field site on / / from __________ to __________ (local time)

Location of LIDAR:

Direction to source __________ Range to source __________ km

Laser inclination (+ angle is up, horizontal is 0°) __________

Source type and official designation:

Plume characteristics (color, shape, steam present, etc.):

Wind speed: begin ______ km/hr end ______ km/hr Wind direction: begin ______ end ______

Air temperature: begin ______ °C end ______ C Relative humidity: begin ______ %, end ______ %

Barometer: begin ______ end ______ Visibility: begin ______ km end ______ km

Cloud cover: begin ______ end ______

Data records made in field (tapes, printouts, photo's, etc.):

MAGNETIC TAPES

tape#	track#	files

OPERATOR'S SIGNATURE: __________ DATE: ______

WITNESS SIGNATURE: __________ DATE: ______

LIDAR OPERATOR'S NOTES

(Include position of laser beam within plume-- attached plume, etc.)

LIDAR FUNCTION VERIFICATION

Source optical generator [ ] screens [ ]

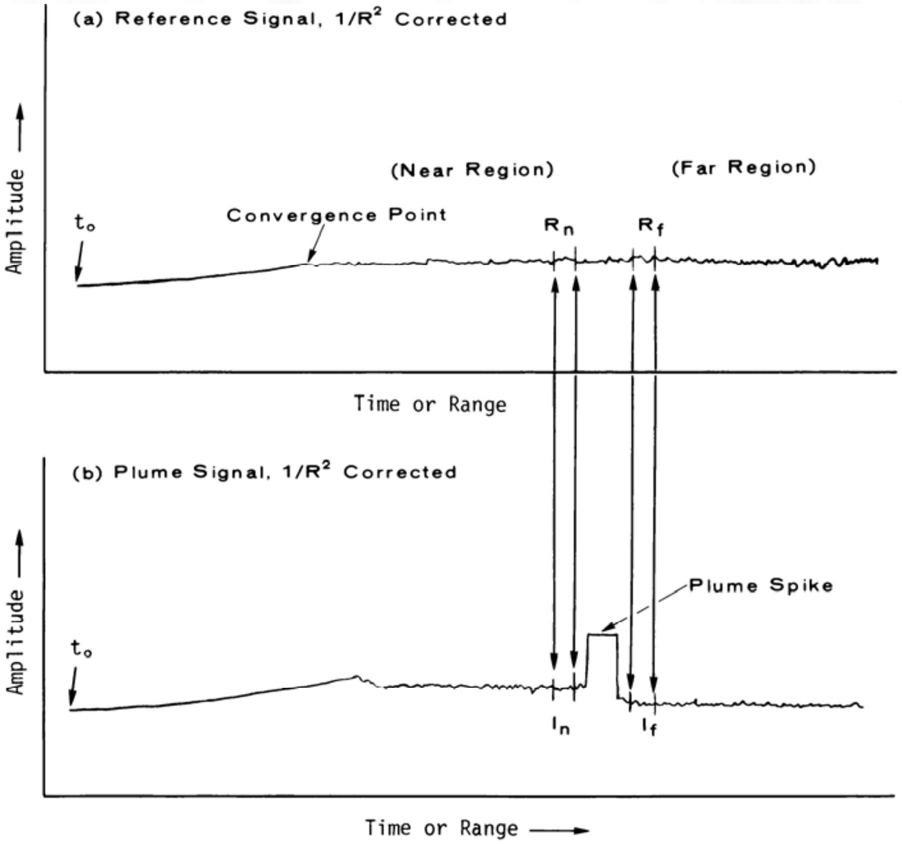
Date of last calibration ______ This test recorded on tape# ______ track# ______

	1	2	3	4	5	6	7	8
Calibrated opacity								
Calculated opacity								
Recorded on file								

OPERATOR'S SIGNATURE: __________ DATE: ______

WITNESS SIGNATURE: __________ DATE: ______

Figure AM1-II Lidar Log Of Operations



(a) Reference signal, 1/R^2-corrected. This reference signal is for plume signal (b). R_n, R_f are chosen to coincide with I_n, I_f.

(b) Plume signal, 1/R^2-corrected. The plume spike and the decrease in the backscatter signal amplitude in the far region are due to the opacity of the plume. I_n, I_f are chosen as indicated in Section 2.6.

Figure AM1-III. Plots of Lidar Backscatter Signals

2.6 Opacity Calculation and Data Analysis. Referring to the reference signal and plume signal in Figure AM1-III, the measured opacity (O_p) in percent for each lidar measurement is calculated using Equation AM1-2. ($O_p = 1 - T_p$; T_p is the plume transmittance.)

$$O_p = (100\%) \left[1 - \left(\frac{I_f}{R_f} \frac{R_n}{I_n} \right)^{\frac{1}{2}} \right], \qquad \text{(AM1-2)}$$

Where:

I_n = near-region pick interval signal amplitude, plume signal, $1/R^2$ corrected,
I_f = far-region pick interval signal amplitude, plume signal, $1/R^2$ corrected,
R_n = near-region pick interval signal amplitude, reference signal, $1/R^2$ corrected, and
R_f = far-region pick interval signal amplitude, reference signal, $1/R^2$ corrected.

The $1/R^2$ correction to the plume and reference signal amplitudes is made by multiplying the amplitude for each successive sample interval from the time reference, by the square of the lidar time (or range) associated with that sample interval [Reference 5.1].

The first step in selecting the pick intervals for Equation AM1–2 is to divide the plume signal amplitude by the reference signal amplitude at the same respective ranges to obtain a "normalized" signal. The pick intervals selected using this normalized signal, are a minimum of 15 m (100 nanoseconds) in length and consist of at least 5 contiguous sample intervals. In addition, the following criteria, listed in order of importance, govern pick interval selection. (1) The intervals shall be in a region of the normalized signal where the reference signal meets the requirements of section 2.3 and is everywhere greater than zero. (2) The intervals (near and far) with the minimum average amplitude are chosen. (3) If more than one interval with the same minimum average amplitude is found, the interval closest to the plume is chosen. (4) The standard deviation, S_o, for the calculated opacity shall be 8% or less. (S_o is calculated by Equation AM1–7).

If S_o is greater than 8%, then the far pick interval shall be changed to the next interval of minimal average amplitude. If S_o is still greater than 8%, then this procedure is repeated for the far pick interval. This procedure may be repeated once again for the near pick interval, but if S_o remains greater than 8%, the plume signal shall be discarded.

The reference signal pick intervals, R_n and R_f, must be chosen over the same time interval as the plume signal pick intervals, I_n and I_f, respectively [Figure AM1–III]. Other methods of selecting pick intervals may be used if they give equivalent results. Field-oriented examples of pick interval selection are available in Reference 5.1.

The average amplitudes for each of the pick intervals, I_n, I_f, R_n, R_f, shall be calculated by averaging the respective individual amplitudes of the sample intervals from the plume signal and the associated reference signal each corrected for $1/R^2$. The amplitude of I_n shall be calculated according to Equation (AM–3).

$$I_n = \frac{1}{m} \sum_{i=1}^{m} I_{ni}, \qquad \text{(AM1-3)}$$

Where:

I_{ni} = the amplitude of the ith sample interval (near-region),
Σ = sum of the individual amplitudes for the sample intervals,
m = number of sample intervals in the pick interval, and
I_n = average amplitude of the near-region pick interval.

Similarly, the amplitudes for I_f, R_n, and R_f are calculated with the three expressions in Equation (AM1–4).

$$I_f = \frac{1}{m} \sum_{i=1}^{m} I_{fi}, \quad R_n = \frac{1}{m} \sum_{i=1}^{m} R_{ni}, \quad R_f = \frac{1}{m} \sum_{i=1}^{m} R_{fi}. \qquad \text{(AM1-4)}$$

The standard deviation, S_{In}, of the set of amplitudes for the near-region pick interval, I_n, shall be calculated using Equation (AM1–5).

Similarly, the standard deviations S_{If}, S_{Rn}, and S_{Rf} are calculated with the three expressions in Equation (AM1–6).

$$S_{In} = \left[\sum_{i=1}^{m} \frac{(I_{ni} - I_n)^2}{(m-1)} \right]^{\frac{1}{2}} . \qquad S_{If} = \left[\sum_{i=1}^{m} \frac{(I_{fi} - I_f)^2}{(m-1)} \right]^{\frac{1}{2}}$$

(AM1-5)

$$S_{Rn} = \left[\sum_{i=1}^{m} \frac{(R_{ni} - R_n)^2}{(m-1)} \right]^{\frac{1}{2}} ,$$

$$S_{Rf} = \left[\sum_{i=1}^{m} \frac{(R_{fi} - R_f)^2}{(m-1)} \right]^{\frac{1}{2}} .$$

(AM1-6)

The standard deviation, S_o, for each associated opacity value, O_p, shall be calculated using Equation (AM1–7).

$$S_o = \frac{(100\%)}{2} \left(\frac{I_f}{R_f} \frac{R_n}{I_n} \right)^{\frac{1}{2}} \left[\frac{S_{In}^2}{I_n^2} + \frac{S_{If}^2}{I_f^2} + \frac{S_{Rn}^2}{R_n^2} + \frac{S_{Rf}^2}{R_f^2} \right]^{\frac{1}{2}}$$

(AM1-7)

The calculated values of I_n, I_f, R_n, R_f, S_{In}, S_{If}, S_{Rn}, S_{Rf}, O_p, and S_o should be recorded. Any plume signal with an S_o greater than 8% shall be discarded.

2.6.1 Azimuth Angle Correction. If the azimuth angle correction to opacity specified in this section is performed, then the elevation angle correction specified in section 2.6.2 shall not be performed. When opacity is measured in the residual region of an attached steam plume, and the lidar line-of-sight is not perpendicular to the plume, it may be necessary to correct the opacity measured by the lidar to obtain the opacity that would be measured on a path perpendicular to the plume. The following method, or any other method which produces equivalent results, shall be used to determine the need for a correction, to calculate the correction, and to document the point within the plume at which the opacity was measured.

Figure AM1–IV(b) shows the geometry of the opacity correction. L′ is the path through the plume along which the opacity measurement is made. P′ is the path perpendicular to the plume at the same point. The angle ε is the angle between L′ and the plume center line. The angle (π/2-ε), is the angle between the L′ and P′. The measured opacity, O_p, measured along the path L′ shall be corrected to obtain the corrected opacity, O_{pc}, for the path P′, using Equation (AM1–8).

$$O_{pc} = (100\%) \left[1 - (1 - 0.01\, O_p)^{\text{Cos } (\pi/2-\varepsilon)} \right]$$

$$= (100\%) \left[1 - (1 - 0.01\, O_p)^{\text{Sin } \varepsilon} \right] \qquad \text{(AM1-8)}$$

The correction in Equation (AM1–8) shall be performed if the inequality in Equation (AM1–9) is true.

$$\varepsilon \geq \mathrm{Sin}^{-1} \left[\frac{\ln(101 - O_p)}{\ln(100 - O_p)} \right] \qquad \text{(AM1-9)}$$

Figure AM1–IV(a) shows the geometry used to calculate ε and the position in the plume at which the lidar measurement is made. This analysis assumes that for a given lidar measurement, the range from the lidar to the plume, the elevation angle of the lidar from the horizontal plane, and the azimuth angle of the lidar from an arbitrary fixed reference in the horizontal plane can all be obtained directly.

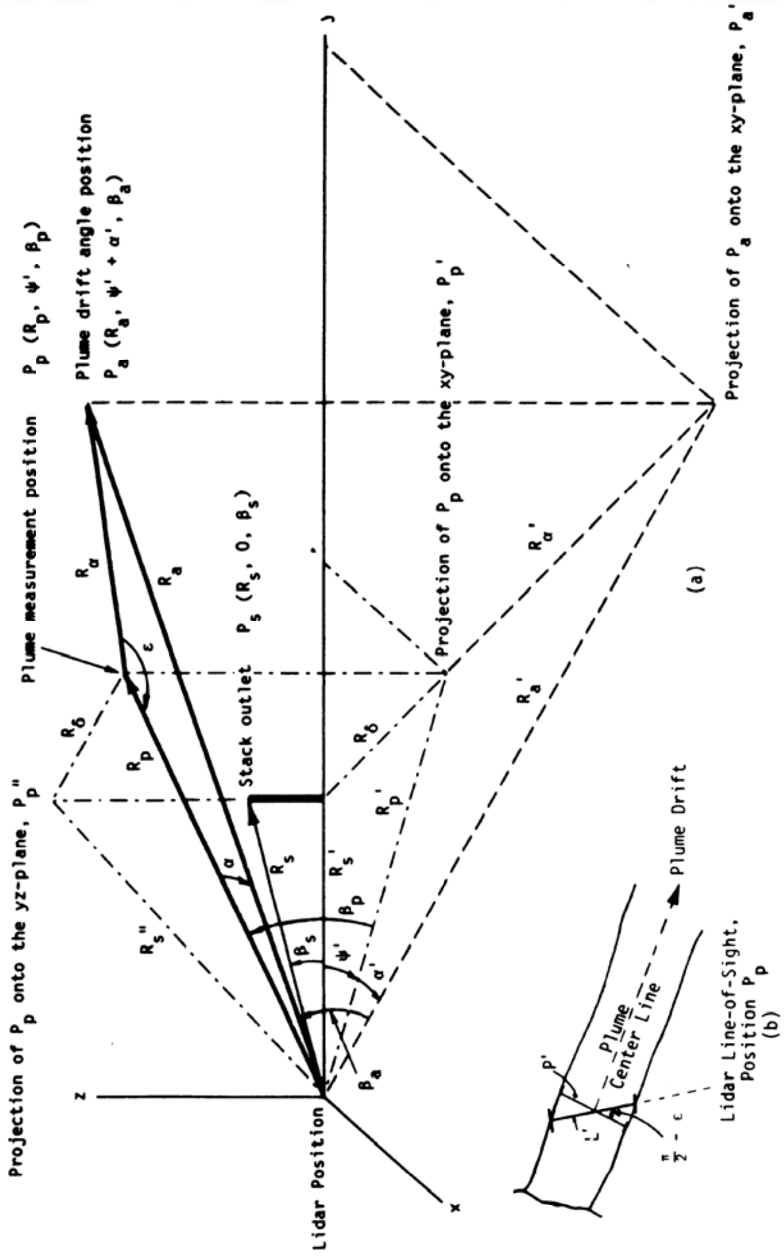


Figure AMI - IV. Correction in Opacity for Drift of the Residual Region of an Attached Steam Plume.

R_s = range from lidar to source*

β_s = elevation angle of R_s*

R_p = range from lidar to plume at the opacity measurement point*

β_p = elevation angle of R_p*

R_a = range from lidar to plume at some arbitrary point, P_a, so the drift angle of the plume can be determined*

β_a = elevation angle of R_a*
α = angle between R_p and R_a
R'_s = projection of R_s in the horizontal plane
R'_p = projection of R_p in the horizontal plane
R'_a = projection of R_a in the horizontal plane
ψ' = angle between R'_s and R'_p*
α' = angle between R'_p and R'_a*
$R\leq$ = distance from the source to the opacity measurement point projected in the horizontal plane
$R\theta$ = distance from opacity measurement point P_p to the point in the plume P_a.

$$0_{pc} = 1 - (1 - 0_p)^{\mathrm{Cos}\,(\pi/2-\varepsilon)} = 1 - (1 - 0_p)^{\mathrm{Sin}\,\varepsilon} \qquad \text{(AMI-8)}$$

The correction angle ε shall be determined using Equation AM1–10.

Where:

$\alpha = \mathrm{Cos}^{-1}\,(\mathrm{Cos}\beta_p\,\mathrm{Cos}\beta_a\,\mathrm{Cos}\alpha' + \mathrm{Sin}\beta_p\,\mathrm{Sin}\beta_a)$, and

$R\theta = (R_p2 + R_a2 - 2\,R_p\,R_a\,\mathrm{Cos}\alpha)^{1/2}$

$R\leq$, the distance from the source to the opacity measurement point projected in the horizontal plane, shall be determined using Equation AM1–11.

$$R_\delta = (R'^{2}_{s} + R'^{2}_{p} - 2R'_sR'_p\mathrm{Cos}\psi')^{1/2}, \qquad \text{(AM1-11)}$$

Where:

$R'_s = R_s\,\mathrm{Cos}\,\beta_s$, and
$R'_p = R_p\,\mathrm{Cos}\,\beta_p$.

In the special case where the plume centerline at the opacity measurement point is horizontal, parallel to the ground, Equation AM1–12 may be used to determine ε instead of Equation AM1–10.

$$\varepsilon = \mathrm{Cos}^{-1}\left[\frac{R_p^{\,2} + R_\delta^{\,2} - R''^{2}_{s}}{2\,R_p\,R_\delta}\right] \qquad \text{(AM1-12)}$$

Where:

$R''_s = (R'^2_s + R_p^2\mathrm{Sin}^2\beta_p)^{1/2}$.

If the angle ε is such that $\varepsilon \leq 30^\circ$ or $\varepsilon \geq 150^\circ$, the azimuth angle correction shall not be performed and the associated opacity value shall be discarded.

2.6.2 Elevation Angle Correction. An individual lidar-measured opacity, O_p, shall be corrected for elevation angle if the laser elevation or inclination angle, β_p [Figure AM1–V], is greater than or equal to the value calculated in Equation AM1–13.

$$\beta_p \geq \mathrm{Cos}^{-1}\left[\frac{\ln\,(101 - 0_p)}{\ln\,(100 - 0_p)}\right] \qquad \text{(AM1-13)}$$

The measured opacity, O_p, along the lidar path L, is adjusted to obtain the corrected opacity, O_{pc}, for the actual plume (horizontal) path, P, by using Equation (AM1–14).

---

*Obtained directly from lidar. These values should be recorded.

$$O_{pc} = (100\%)\left[1-\left(1-0.01\,O_p\right)^{\mathrm{Cos}\beta} p\right], \qquad (\mathrm{AM1\text{-}14})$$

Where:

β_p = lidar elevation or inclination angle,

O_p = measured opacity along path L, and

O_{pc} = corrected opacity for the actual plume thickness P.

The values for β_p, O_p and O_{pc} should be recorded.

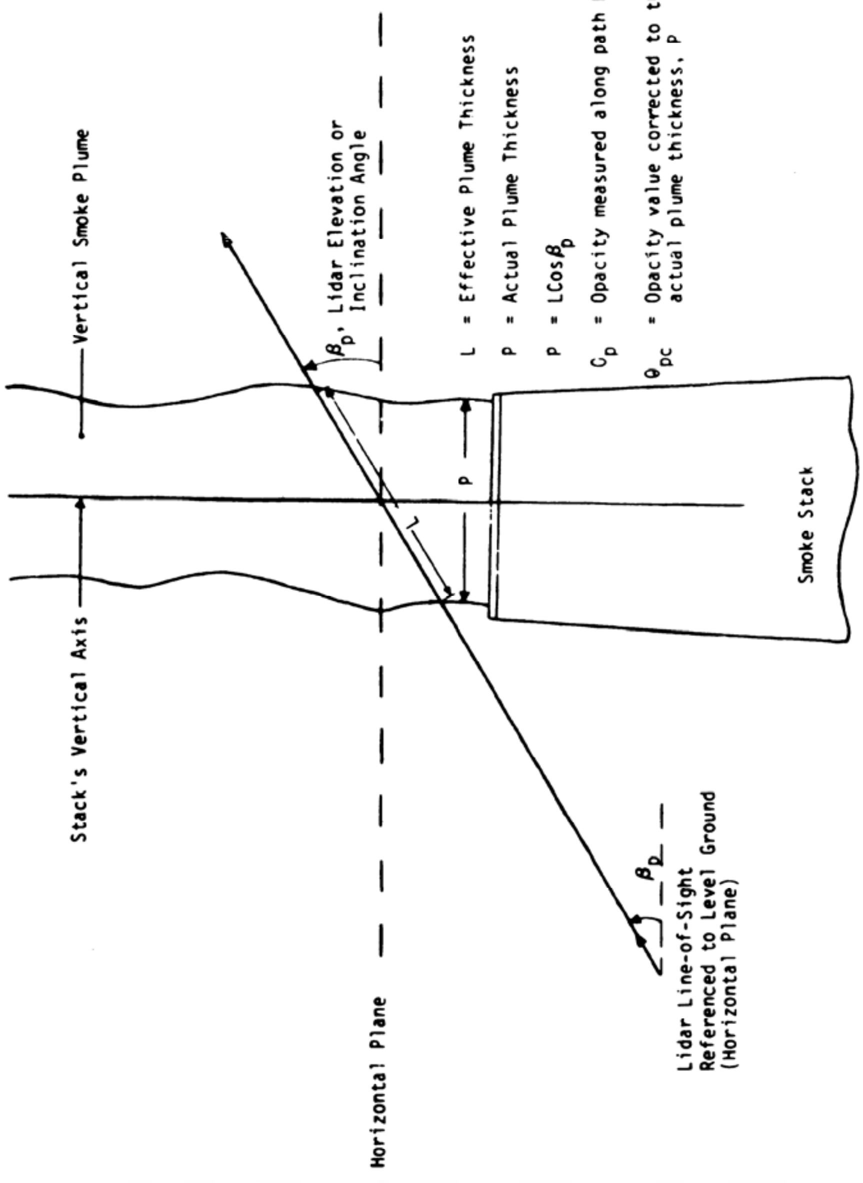


Figure AM1-V. Elevation Angle Correction for Vertical Plumes.

2.6.3 Determination of Actual Plume Opacity. Actual opacity of the plume shall be determined by Equation AM1–15.

$$O_{pa} = O_{pc} - [2 S_o + 5\%]. \qquad \text{(AM1-15)}$$

2.6.4 Calculation of Average Actual Plume Opacity. The average of the actual plume opacity, O_{pa}, shall be calculated as the average of the consecutive individual actual opacity values, O_{pa}, by Equation AM1–16.

$$\bar{O}_{pa} = \frac{1}{n} \sum_{k=1}^{n} (O_{pa})_k \,, \qquad \text{(AM1-16)}$$

Where:

$(O_{pa})_k$ = the kth actual opacity value in an averaging interval containing n opacity values; k is a summing index.

Σ = the sum of the individual actual opacity values.

n = the number of individual actual opacity values contained in the averaging interval.

O_{pa} = average actual opacity calculated over the averaging interval.

3. Lidar Performance Verification

The lidar shall be subjected to two types of performance verifications that shall be performed in the field. The annual calibration, conducted at least once a year, shall be used to directly verify operation and performance of the entire lidar system. The routine verification, conducted for each emission source measured, shall be used to insure proper performance of the optical receiver and associated electronics.

3.1 Annual Calibration Procedures. Either a plume from a smoke generator or screen targets shall be used to conduct this calibration.

If the screen target method is selected, five screens shall be fabricated by placing an opaque mesh material over a narrow frame (wood, metal extrusion, etc.). The screen shall have a surface area of at least one square meter. The screen material should be chosen for precise optical opacities of about 10, 20, 40, 60, and 80%. Opacity of each target shall be optically determined and should be recorded. If a smoke generator plume is selected, it shall meet the requirements of section 3.3 of Reference Method 9. This calibration shall be performed in the field during calm (as practical) atmospheric conditions. The lidar shall be positioned in accordance with section 2.1.

The screen targets must be placed perpendicular to and coincident with the lidar line-of-sight at sufficient height above the ground (suggest about 30 ft) to avoid ground-level dust contamination. Reference signals shall be obtained just prior to conducting the calibration test.

The lidar shall be aimed through the center of the plume within 1 stack diameter of the exit, or through the geometric center of the screen target selected. The lidar shall be set in operation for a 6-minute data run at a nominal pulse rate of 1 pulse every 10 seconds. Each backscatter return signal and each respective opacity value obtained from the smoke generator transmissometer, shall be obtained in temporal coincidence. The data shall be analyzed and reduced in accordance with section 2.6 of this method. This calibration shall be performed for 0% (clean air), and at least five other opacities (nominally 10, 20, 40, 60, and 80%).

The average of the lidar opacity values obtained during a 6-minute calibration run shall be calculated and should be recorded. Also the average of the opacity values obtained from the smoke generator transmissometer for the same 6-minute run shall be calculated and should be recorded.

Alternate calibration procedures that do not meet the above requirements but produce equivalent results may be used.

3.2 Routine Verification Procedures. Either one of two techniques shall be used to conduct this verification. It shall be performed at least once every 4 hours for each emission source measured. The following parameters shall be directly verified.

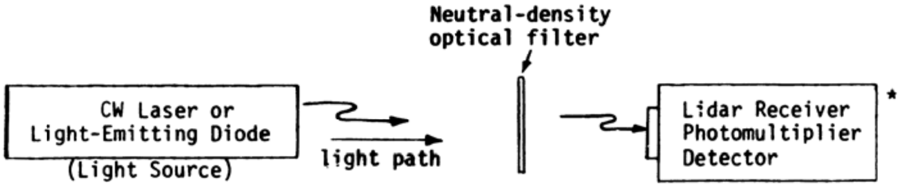
1) The opacity value of 0% plus a minimum of 5 (nominally 10, 20, 40, 60, and 80%) opacity values shall be verified through the PMT detector and data processing electronics.

2) The zero-signal level (receiver signal with no optical signal from the source present) shall be inspected to insure that no spurious noise is present in the signal. With the entire lidar receiver and analog/digital electronics turned on and adjusted for normal operating performance, the following procedures shall be used for Techniques 1 and 2, respectively.

3.2.1 Procedure for Technique 1. This test shall be performed with no ambient or stray light reaching the PMT detector. The narrow band filter (694.3 nanometers peak) shall be removed from its position in front of the PMT detector. Neutral density filters of nominal opacities of 10, 20, 40, 60, and 80% shall be used. The recommended test configuration is depicted in Figure AM1–VI.

(a) Zero-Signal Level Test

(b) Clear-Air or 0% Opacity Test



(c) Optical Filter Test (simulated opacity values)

*Tests shall be performed with no ambient or stray light reaching the detector.

Figure AM1-VI. Test Configuration for Technique 1.

The zero-signal level shall be measured and should be recorded, as indicated in Figure AM1–VI(a). This simulated clear-air or 0% opacity value shall be tested in using the selected light source depicted in Figure AM1–VI(b).

The light source either shall be a continuous wave (CW) laser with the beam mechanically chopped or a light emitting diode controlled with a pulse generator (rectangular pulse). (A laser beam may have to be attenuated so as not to saturate the PMT detector). This signal level shall be measured and should be recorded. The opacity value is calculated by taking two pick intervals [Section 2.6] about 1 microsecond apart in time and using Equation (AM1–2) setting the ratio $R_n/R_f = 1$. This calculated value should be recorded.

The simulated clear-air signal level is also employed in the optical test using the neutral density filters. Using the test configuration in Figure AM1–VI(c), each neutral density filter shall be separately placed into the light path from the light source to the PMT detector. The signal level shall be measured and should be recorded. The opacity value for each filter is calculated by taking the signal level for that respective filter (I_f), dividing it by the 0% opacity signal level (I_n) and performing the remainder of the calculation by Equation (AM1–2) with $R_n/R_f = 1$. The calculated opacity value for each filter should be recorded.

The neutral density filters used for Technique 1 shall be calibrated for actual opacity with accuracy of ±2% or better. This calibration shall be done monthly while the filters are in use and the calibrated values should be recorded.

3.2.2 Procedure for Technique 2. An optical generator (built-in calibration mechanism) that contains a light-emitting diode (red light for a lidar containing a ruby laser) is used. By injecting an optical signal into the lidar receiver immediately ahead of the PMT detector, a backscatter signal is simulated. With the entire lidar receiver electronics turned on and adjusted for normal operating performance, the optical generator is turned on and the simulation signal (corrected for $1/R^2$) is selected with no plume spike signal and with the opacity value equal to 0%. This simulated clear-air atmospheric return signal is displayed on the system's video display. The lidar operator then makes any fine adjustments that may be necessary to maintain the system's normal operating range.

The opacity values of 0% and the other five values are selected one at a time in any order. The simulated return signal data should be recorded. The opacity value shall be calculated. This measurement/calculation shall be performed at least three times for each selected opacity value. While the order is not important, each of the opacity values from the optical generator shall be verified. The calibrated optical generator opacity value for each selection should be recorded.

The optical generator used for Technique 2 shall be calibrated for actual opacity with an accuracy of ±1% or better. This calibration shall be done monthly while the generator is in use and calibrated value should be recorded.

Alternate verification procedures that do not meet the above requirements but produce equivalent results may be used.

3.3 Deviation. The permissible error for the annual calibration and routine verification are:

3.3.1 Annual Calibration Deviation.

3.3.1.1 Smoke Generator. If the lidar-measured average opacity for each data run is not within ±5% (full scale) of the respective smoke generator's average opacity over the range of 0% through 80%, then the lidar shall be considered out of calibration.

3.3.1.2 Screens. If the lidar-measured average opacity for each data run is not within ±3% (full scale) of the laboratory-determined opacity for each respective simulation screen target over the range of 0% through 80%, then the lidar shall be considered out of calibration.

3.3.2 Routine Verification Error. If the lidar-measured average opacity for each neutral density filter (Technique 1) or optical generator selection (Technique 2) is not within ±3% (full scale) of the respective laboratory calibration value then the lidar shall be considered non-operational.

4. Performance/Design Specification for Basic Lidar System

4.1 Lidar Design Specification. The essential components of the basic lidar system are a pulsed laser (transmitter), optical receiver, detector, signal processor, recorder, and an aiming device that is used in aiming the lidar transmitter and receiver. Figure AM1–VII shows a functional block diagram of a basic lidar system.

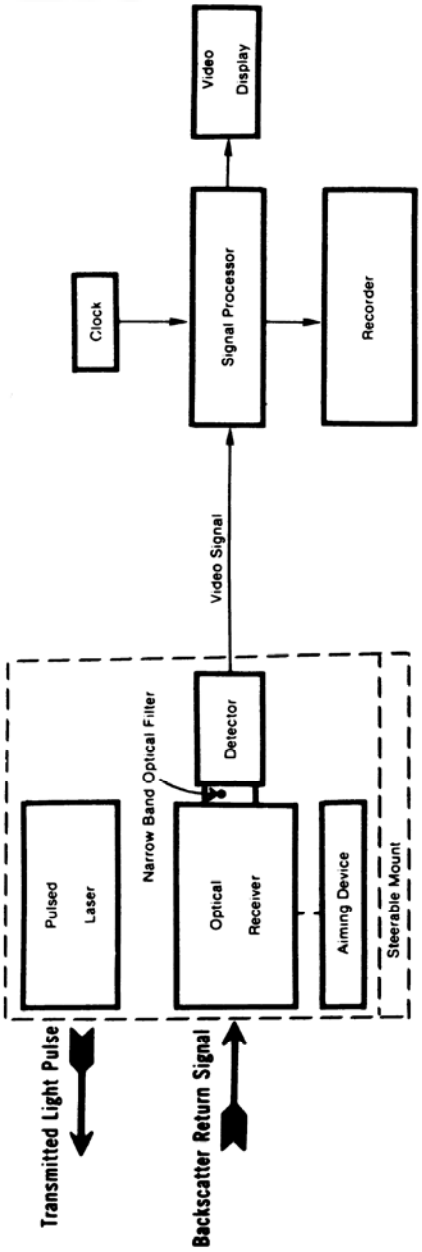


Figure AM1-VII. Functional Block Diagram of a Basic Lidar System

4.2 Performance Evaluation Tests. The owner of a lidar system shall subject such a lidar system to the performance verification tests described in section 3, prior to first use of this method. The annual calibration shall be performed for three separate, complete runs and the results of each should be recorded. The requirements of section 3.3.1 must be fulfilled for each of the three runs.

Once the conditions of the annual calibration are fulfilled the lidar shall be subjected to the routine verification for three separate complete runs. The requirements of section 3.3.2 must be fulfilled for each of the three runs and the results should be recorded. The Administrator may request that the results of the performance evaluation be submitted for review.

5. References

5.1 The Use of Lidar for Emissions Source Opacity Determination, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA–330/1–79–003–R, Arthur W. Dybdahl, current edition [NTIS No. PB81–246662].

5.2 Field Evaluation of Mobile Lidar for the Measurement of Smoke Plume Opacity, U.S. Environmental Protection Agency, National Enforcement Investigations Center, Denver, CO. EPA/NEIC-TS-128, February 1976.

5.3 Remote Measurement of Smoke Plume Transmittance Using Lidar, C. S. Cook, G. W. Bethke, W. D. Conner (EPA/RTP). Applied Optics 11, pg 1742. August 1972.

5.4 Lidar Studies of Stack Plumes in Rural and Urban Environments, EPA–650/4–73–002, October 1973.

5.5 American National Standard for the Safe Use of Lasers ANSI Z 136.1–176, March 8, 1976.

5.6 U.S. Army Technical Manual TB MED 279, Control of Hazards to Health from Laser Radiation, February 1969.

5.7 Laser Institute of America Laser Safety Manual, 4th Edition.

5.8 U.S. Department of Health, Education and Welfare, Regulations for the Administration and Enforcement of the Radiation Control for Health and Safety Act of 1968, January 1976.

5.9 Laser Safety Handbook, Alex Mallow, Leon Chabot, Van Nostrand Reinhold Co., 1978.

METHOD 10—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES (INSTRUMENTAL ANALYZER PROCEDURE)

1.0 Scope and Application

What is Method 10?

Method 10 is a procedure for measuring carbon monoxide (CO) in stationary source emissions using a continuous instrumental analyzer. Quality assurance and quality control requirements are included to assure that you, the tester, collect data of known quality. You must document your adherence to these specific requirements for equipment, supplies, sample collection and analysis, calculations, and data analysis. This method does not completely describe all equipment, supplies, and sampling and analytical procedures you will need but refers to other methods for some of the details. Therefore, to obtain reliable results, you should also have a thorough knowledge of these additional test methods which are found in appendix A to this part:

(a) Method 1—Sample and Velocity Traverses for Stationary Sources.

(b) Method 4—Determination of Moisture Content in Stack Gases.

(c) Method 7E—Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyzer Procedure).

1.1 Analytes. What does this method determine? This method measures the concentration of carbon monoxide.

Analyte	CAS No.	Sensitivity
CO	630–08–0	Typically <2% of Calibration Span.

1.2 Applicability. When is this method required? The use of Method 10 may be required by specific New Source Performance Standards, State Implementation Plans, and permits where CO concentrations in stationary source emissions must be measured, either to determine compliance with an applicable emission standard or to conduct performance testing of a continuous emission monitoring system (CEMS). Other regulations may also require the use of Method 10.

1.3 Data Quality Objectives. Refer to section 1.3 of Method 7E.

2.0 Summary of Method

In this method, you continuously or intermittently sample the effluent gas and convey the sample to an analyzer that measures the concentration of CO. You must meet the performance requirements of this method to validate your data.

3.0 Definitions

Refer to section 3.0 of Method 7E for the applicable definitions.

4.0 Interferences

Substances having a strong absorption of infrared energy may interfere to some extent in some analyzers. Instrumental correction may be used to compensate for the interference. You may also use silica gel and ascarite traps to eliminate the interferences. If this option is used, correct the measured gas volume for the carbon dioxide (CO_2) removed in the trap.

5.0 Safety

Refer to section 5.0 of Method 7E.

6.0 Equipment and Supplies

What do I need for the measurement system?

6.1 Continuous Sampling. Figure 7E–1 of Method 7E is a schematic diagram of an acceptable measurement system. The components are the same as those in sections 6.1 and 6.2 of Method 7E, except that the CO analyzer described in section 6.2 of this method must be used instead of the analyzer described in section 6.2 of Method 7E. You must follow the noted specifications in section 6.1 of Method 7E except that the requirements to use stainless steel, Teflon, or non-reactive glass filters do not apply. Also, a heated sample line is not required to transport dry gases or for systems that measure the CO concentration on a dry basis.

6.2 Integrated Sampling.

6.2.1 Air-Cooled Condenser or Equivalent. To remove any excess moisture.

6.2.2 Valve. Needle valve, or equivalent, to adjust flow rate.

6.2.3 Pump. Leak-free diaphragm type, or equivalent, to transport gas.

6.2.4 Rate Meter. Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per minute (0.035 cfm).

6.2.5 Flexible Bag. Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft^3). (Verify through the manufacturer that the Tedlar alternative is suitable for CO and make this verified information available for inspection.) Leak-test the bag in the laboratory before using by evacuating with a pump followed by a dry gas meter. When the evacuation is complete, there should be no flow through the meter.

6.2.6 Sample Tank. Stainless steel or aluminum tank equipped with a pressure indicator with a minimum volume of 4 liters.

6.3 What analyzer must I use? You must use an instrument that continuously measures CO in the gas stream and meets the specifications in section 13.0. The dual-range analyzer provisions in section 6.2.8.1 of Method 7E apply.

7.0 Reagents and Standards

7.1 Calibration Gas. What calibration gases do I need? Refer to section 7.1 of Method 7E for the calibration gas requirements.

7.2 Interference Check. What additional reagents do I need for the interference check? Use the appropriate test gases listed in Table 7E–3 of Method 7E (i.e., potential interferents, as identified by the instrument manufacturer) to conduct the interference check.

8.0 Sample Collection, Preservation, Storage, and Transport

Emission Test Procedure

8.1 Sampling Site and Sampling Points. You must follow section 8.1 of Method 7E.

8.2 Initial Measurement System Performance Tests. You must follow the procedures in section 8.2 of Method 7E. If a dilution-type measurement system is used, the special considerations in section 8.3 of Method 7E also apply.

8.3 Interference Check. You must follow the procedures of section 8.2.7 of Method 7E.

8.4 Sample Collection.

8.4.1 Continuous Sampling. You must follow the procedures of section 8.4 of Method 7E.

8.4.2 Integrated Sampling. Evacuate the flexible bag or sample tank. Set up the equipment as shown in Figure 10–1 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak-free. Sample at a rate proportional to the stack velocity. If needed, the CO_2 content of the gas may be determined by using the Method 3 integrated sample procedures, or by weighing an ascarite CO_2 removal tube used and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube. Data may be recorded on a form similar to Table 10–1. If a sample tank is used for sample collection, follow procedures similar to those in sections 8.1.2, 8.2.3, 8.3, and 12.4 of Method 25 as appropriate to prepare the tank, conduct the sampling, and correct the measured sample concentration.

8.5 Post-Run System Bias Check, Drift Assessment, and Alternative Dynamic Spike Procedure. You must follow the procedures in sections 8.5 and 8.6 of Method 7E.

9.0 Quality Control

Follow the quality control procedures in section 9.0 of Method 7E.

10.0 Calibration and Standardization

Follow the procedures for calibration and standardization in section 10.0 of Method 7E.

11.0 Analytical Procedures

Because sample collection and analysis are performed together (see section 8), additional discussion of the analytical procedure is not necessary.

12.0 Calculations and Data Analysis

You must follow the procedures for calculations and data analysis in section 12.0 of Method 7E, as applicable, substituting CO for NO_X as applicable.

12.1 Concentration Correction for CO_2 Removal. Correct the CO concentration for CO_2 removal (if applicable) using Eq. 10–1.

$$C_{Avg} = C_{CO\ stack}\left(1 - F_{CO2}\right)$$

Where:

C_{Avg} = Average gas concentration for the test run, ppm.

$C_{CO\ stack}$ = Average unadjusted stack gas CO concentration indicated by the data recorder for the test run, ppmv.

F_{CO2} = Volume fraction of CO_2 in the sample, i.e., percent CO_2 from Orsat analysis divided by 100.

13.0 Method Performance

The specifications for analyzer calibration error, system bias, drift, interference check, and alternative dynamic spike procedure are the same as in section 13.0 of Method 7E.

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

The dynamic spike procedure and the manufacturer stability test are the same as in sections 16.1 and 16.3 of Method 7E

17.0 References

1. "EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards— September 1997 as amended, EPA-600/R-97/121

18.0 Tables, Diagrams, Flowcharts, and Validation Data

Figure 10-1. Integrated Gas Sampling Train.

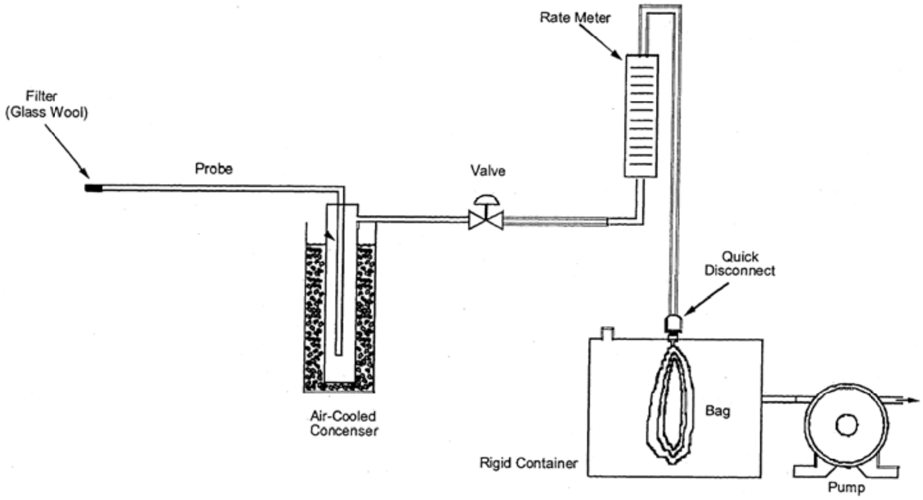


TABLE 10–1—FIELD DATA

[Integrated sampling]

Location: Date:

Test: Operator:

Clock Time	Rotameter Reading liters/min (cfm)	Comments

TABLE 10–1—FIELD DATA—Continued

[Integrated sampling]

METHOD 10A—DETERMINATION OF CARBON MONOXIDE EMISSIONS IN CERTIFYING CONTINUOUS EMISSION MONITORING SYSTEMS AT PETROLEUM REFINERIES

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 4, and Method 5.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Carbon monoxide (CO)	630–08–0	3 ppmv

1.2 Applicability. This method is applicable for the determination of CO emissions at petroleum refineries. This method serves as the reference method in the relative accuracy test for nondispersive infrared (NDIR) CO continuous emission monitoring systems (CEMS) that are required to be installed in petroleum refineries on fluid catalytic cracking unit catalyst regenerators (§ 60.105(a)(2) of this part).

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

An integrated gas sample is extracted from the stack, passed through an alkaline permanganate solution to remove sulfur oxides and nitrogen oxides, and collected in a Tedlar or equivalent bag. (Verify through the manufacturer that the Tedlar alternative is suitable for NO and make this verified information available for inspection.) The CO concentration in the sample is measured spectrophotometrically using the reaction of CO with *p*-sulfaminobenzoic acid.

3.0 Definitions [Reserved]

4.0 Interferences

Sulfur oxides, nitric oxide, and other acid gases interfere with the colorimetric reaction. They are removed by passing the sampled gas through an alkaline potassium permanganate scrubbing solution. Carbon dioxide (CO_2) does not interfere, but, because it is removed by the scrubbing solution, its concentration must be measured independently and an appropriate volume correction made to the sampled gas.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method. The analyzer users manual should be consulted for specific precautions to be taken with regard to the analytical procedure.

5.2 Corrosive reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water for at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burns as thermal burns.

5.2.1 Sodium Hydroxide (NaOH). Causes severe damage to eyes and skin. Inhalation causes irritation to nose, throat, and lungs.

Reacts exothermically with limited amounts of water.

6.0 Equipment and Supplies

6.1 Sample Collection. The sampling train shown in Figure 10A–1 is required for sample collection. Component parts are described below:

6.1.1 Probe. Stainless steel, sheathed Pyrex glass, or equivalent, equipped with a glass wool plug to remove particulate matter.

6.1.2 Sample Conditioning System. Three Greenburg-Smith impingers connected in series with leak-free connections.

6.1.3 Pump. Leak-free pump with stainless steel and Teflon parts to transport sample at a flow rate of 300 ml/min (0.01 ft^3/min) to the flexible bag.

6.1.4 Surge Tank. Installed between the pump and the rate meter to eliminate the pulsation effect of the pump on the rate meter.

6.1.5 Rate Meter. Rotameter, or equivalent, to measure flow rate at 300 ml/min (0.01 ft^3/min). Calibrate according to section 10.2.

6.1.6 Flexible Bag. Tedlar, or equivalent, with a capacity of 10 liters (0.35 ft^3) and equipped with a sealing quick-connect plug. The bag must be leak-free according to section 8.1. For protection, it is recommended that the bag be enclosed within a rigid container.

6.1.7 Sample Tank. Stainless steel or aluminum tank equipped with a pressure indicator with a minimum volume of 10 liters.

6.1.8 Valves. Stainless-steel needle valve to adjust flow rate, and stainless-steel 3-way valve, or equivalent.

6.1.9 CO_2 Analyzer. Fyrite, or equivalent, to measure CO_2 concentration to within 0.5 percent.

6.1.10 Volume Meter. Dry gas meter, capable of measuring the sample volume under calibration conditions of 300 ml/min (0.01 ft^3/min) for 10 minutes.

6.1.11 Pressure Gauge. A water filled U-tube manometer, or equivalent, of about 30 cm (12 in.) to leak-check the flexible bag.

6.2 Sample Analysis.

6.2.1 Spectrophotometer. Single- or double-beam to measure absorbance at 425 and 600 nm. Slit width should not exceed 20 nm.

6.2.2 Spectrophotometer Cells. 1-cm pathlength.

6.2.3 Vacuum Gauge. U-tube mercury manometer, 1 meter (39 in.), with 1-mm divisions, or other gauge capable of measuring pressure to within 1 mm Hg.

6.2.4 Pump. Capable of evacuating the gas reaction bulb to a pressure equal to or less than 40 mm Hg absolute, equipped with coarse and fine flow control valves.

6.2.5 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 1 mm Hg.

6.2.6 Reaction Bulbs. Pyrex glass, 100-ml with Teflon stopcock (Figure 10A–2), leak-free at 40 mm Hg, designed so that 10 ml of the colorimetric reagent can be added and removed easily and accurately. Commercially available gas sample bulbs such as Supelco Catalog No. 2–2161 may also be used.

6.2.7 Manifold. Stainless steel, with connections for three reaction bulbs and the appropriate connections for the manometer and sampling bag as shown in Figure 10A–3.

6.2.8 Pipets. Class A, 10–ml size.

6.2.9 Shaker Table. Reciprocating-stroke type such as Eberbach Corporation, Model 6015. A rocking arm or rotary-motion type shaker may also be used. The shaker must be large enough to accommodate at least six gas sample bulbs simultaneously. It may be necessary to construct a table top extension for most commercial shakers to provide sufficient space for the needed bulbs (Figure 10A–4).

6.2.10 Valve. Stainless steel shut-off valve.

6.2.11 Analytical Balance. Capable of weighing to 0.1 mg.

7.0 Reagents and Standards

Unless otherwise indicated, all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, the best available grade shall be used.

7.1 Sample Collection.

7.1.1 Water. Deionized distilled, to conform to ASTM D 1193–77 or 91, Type 3 (incorporated by reference—see §60.17). If high concentrations of organic matter are not expected to be present, the potassium permanganate test for oxidizable organic matter may be omitted.

7.1.2 Alkaline Permanganate Solution, 0.25 M $KMnO_4$/1.5 M Sodium Hydroxide (NaOH). Dissolve 40 g $KMnO_4$ and 60 g NaOH in approximately 900 ml water, cool, and dilute to 1 liter.

7.2 Sample Analysis.

7.2.1 Water. Same as in section 7.1.1.

7.2.2 1 M Sodium Hydroxide Solution. Dissolve 40 g NaOH in approximately 900 ml of water, cool, and dilute to 1 liter.

7.2.3 0.1 M NaOH Solution. Dilute 50 ml of the 1 M NaOH solution prepared in section 7.2.2 to 500 ml.

7.2.4 0.1 M Silver Nitrate ($AgNO_3$) Solution. Dissolve 8.5 g $AgNO_3$ in water, and dilute to 500 ml.

7.2.5 0.1 M Para-Sulfaminobenzoic Acid (p-SABA) Solution. Dissolve 10.0 g p-SABA in 0.1 M NaOH, and dilute to 500 ml with 0.1 M NaOH.

7.2.6 Colorimetric Solution. To a flask, add 100 ml of 0.1 M p-SABA solution and 100 ml of 0.1 M $AgNO_3$ solution. Mix, and add 50 ml of 1 M NaOH with shaking. The resultant solution should be clear and colorless. This solution is acceptable for use for a period of 2 days.

7.2.7 Standard Gas Mixtures. Traceable to National Institute of Standards and Technology (NIST) standards and containing between 50 and 1000 ppm CO in nitrogen. At least two concentrations are needed to span each calibration range used (Section 10.3). The calibration gases must be certified by the manufacturer to be within 2 percent of the specified concentrations.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Sample Bag or Tank Leak-Checks. While a leak-check is required after bag or sample tank use, it should also be done before the bag or sample tank is used for sample collection. The tank should be leak-checked according to the procedure specified in section 8.1.2 of Method 25. The bag should be leak-checked in the inflated and deflated condition according to the following procedure:

8.1.1 Connect the bag to a water manometer, and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in H_2O). Allow the bag to stand for 60 minutes. Any displacement in the water manometer indicates a leak.

8.1.2 Evacuate the bag with a leakless pump that is connected to the downstream side of a flow indicating device such as a 0- to 100-ml/min rotameter or an impinger containing water. When the bag is completely evacuated, no flow should be evident if the bag is leak-free.

8.2 Sample Collection.

8.2.1 Evacuate and leak check the sample bag or tank as specified in section 8.1. Assemble the apparatus as shown in Figure 10A–1. Loosely pack glass wool in the tip of the probe. Place 400 ml of alkaline permanganate solution in the first two impingers and 250 ml in the third. Connect the pump to the third impinger, and follow this with the surge tank, rate meter, and 3-way valve. Do not connect the bag or sample tank to the system at this time.

8.2.2 Leak-check the sampling system by plugging the probe inlet, opening the 3-way valve, and pulling a vacuum of approximately 250 mm Hg on the system while observing the rate meter for flow. If flow is indicated on the rate meter, do not proceed further until the leak is found and corrected.

8.2.3 Purge the system with sample gas by inserting the probe into the stack and drawing the sample gas through the system at 300 ml/min ±10 percent for 5 minutes. Connect the evacuated bag or sample tank to the system, record the starting time, and sample at a rate of 300 ml/min for 30 minutes, or until the bag is nearly full, or the sample tank reaches ambient pressure. Record the sampling time, the barometric pressure, and the ambient temperature. Purge the system as described above immediately before each sample.

8.2.4 The scrubbing solution is adequate for removing sulfur oxides and nitrogen oxides from 50 liters (1.8 ft^3) of stack gas when the concentration of each is less than 1,000 ppm and the CO_2 concentration is less than 15 percent. Replace the scrubber solution after every fifth sample.

8.3 Carbon Dioxide Measurement. Measure the CO_2 content in the stack to the nearest 0.5 percent each time a CO sample is collected. A simultaneous grab sample analyzed by the Fyrite analyzer is acceptable.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.1	Sampling equipment leak-checks and calibration.	Ensure accuracy and precision of sampling measurements.
10.3	Spectrophotometer calibration	Ensure linearity of spectrophotometer response to standards.

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

NOTE: Maintain a laboratory log of all calibrations.

10.1 Gas Bulb Calibration. Weigh the empty bulb to the nearest 0.1 g. Fill the bulb to the stopcock with water, and again weigh to the nearest 0.1 g. Subtract the tare weight, and calculate the volume in liters to three significant figures using the density of water at the measurement temperature. Record the volume on the bulb. Alternatively, mark an identification number on the bulb, and record the volume in a notebook.

10.2 Rate Meter Calibration. Assemble the system as shown in Figure 10A–1 (the impingers may be removed), and attach a volume meter to the probe inlet. Set the rotameter at 300 ml/min, record the volume meter reading, start the pump, and pull ambient air through the system for 10 minutes. Record the final volume meter reading. Repeat the procedure and average the results to determine the volume of gas that passed through the system.

10.3 Spectrophotometer Calibration Curve.

10.3.1 Collect the standards as described in section 8.2. Prepare at least two sets of three bulbs as standards to span the 0 to 400 or 400 to 1000 ppm range. If any samples span both concentration ranges, prepare a calibration curve for each range using separate reagent blanks. Prepare a set of three bulbs containing colorimetric reagent but no CO to serve as a reagent blank. Analyze each standard and blank according to the sample analysis procedure of section 11.0 Reject the standard set where any of the individual bulb absorbances differs from the set mean by more than 10 percent.

10.3.2 Calculate the average absorbance for each set (3 bulbs) of standards using Equation 10A–1 and Table 10A–1. Construct a graph of average absorbance for each standard against its corresponding concentration. Draw a smooth curve through the points. The curve should be linear over the two concentration ranges discussed in section 13.3.

11.0 Analytical Procedure

11.1 Assemble the system shown in Figure 10A–3, and record the information required in Table 10A–1 as it is obtained. Pipet 10.0 ml of the colorimetric reagent into each gas reaction bulb, and attach the bulbs to the system. Open the stopcocks to the reaction bulbs, but leave the valve to the bag closed. Turn on the pump, fully open the coarse-adjust flow valve, and slowly open the fine-adjust valve until the pressure is reduced to at least 40 mm Hg. Now close the coarse adjust valve, and observe the manometer to be certain that the system is leak-free. Wait a minimum of 2 minutes. If the pressure has increased less than 1 mm Hg, proceed as described below. If a leak is present, find and correct it before proceeding further.

11.2 Record the vacuum pressure (P_v) to the nearest 1 mm Hg, and close the reaction bulb stopcocks. Open the bag valve, and allow the system to come to atmospheric pressure. Close the bag valve, open the pump coarse adjust valve, and evacuate the system again. Repeat this fill/evacuation procedure at least twice to flush the manifold completely. Close the pump coarse adjust valve, open the bag valve, and let the system fill to atmospheric pressure. Open the stopcocks to the reaction bulbs, and let the entire system come to atmospheric pressure. Close the bulb stopcocks, remove the bulbs, record the room temperature and barometric pressure (P_{bar}, to nearest mm Hg), and place the bulbs on the shaker table with their main axis either parallel to or perpendicular to the plane of the table top. Purge the bulb-filling system with ambient air for several minutes between samples. Shake the samples for exactly 2 hours.

11.3 Immediately after shaking, measure the absorbance (A) of each bulb sample at 425 nm if the concentration is less than or equal to 400 ppm CO or at 600 nm if the concentration is above 400 ppm.

NOTE: This may be accomplished with multiple bulb sets by sequentially collecting sets and adding to the shaker at staggered intervals, followed by sequentially removing sets from the shaker for absorbance measurement after the two-hour designated intervals have elapsed.

11.4 Use a small portion of the sample to rinse a spectrophotometer cell several times before taking an aliquot for analysis. If one cell is used to analyze multiple samples, rinse the cell with deionized distilled water several times between samples. Prepare and analyze standards and a reagent blank as described in section 10.3. Use water as the reference. Reject the analysis if the blank absorbance is greater than 0.1. All conditions should be the same for analysis of samples and standards. Measure the absorbances as soon as possible after shaking is completed.

11.5 Determine the CO concentration of each bag sample using the calibration curve for the appropriate concentration range as discussed in section 10.3.

12.0 Calculations and Data Analysis

Carry out calculations retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

12.1 Nomenclature.

A = Sample absorbance, uncorrected for the reagent blank.
A_r = Absorbance of the reagent blank.
A_s = Average sample absorbance per liter, units/liter.
B_w = Moisture content in the bag sample.
C = CO concentration in the stack gas, dry basis, ppm.
C_b = CO concentration of the bag sample, dry basis, ppm.
C_g = CO concentration from the calibration curve, ppm.
F = Volume fraction of CO_2 in the stack.
n = Number of reaction bulbs used per bag sample.
P_b = Barometric pressure, mm Hg.
P_v = Residual pressure in the sample bulb after evacuation, mm Hg.
P_w = Vapor pressure of H_2O in the bag (from Table 10A–2), mm Hg.
V_b = Volume of the sample bulb, liters.
V_r = Volume of reagent added to the sample bulb, 0.0100 liter.

12.2 Average Sample Absorbance per Liter. Calculate A_s for each gas bulb using Equation 10A–1, and record the value in Table 10A–1. Calculate the average A_s for each bag sample, and compare the three values to the average. If any single value differs by more than 10 percent from the average, reject this value, and calculate a new average using the two remaining values.

$$A_s = \frac{(A - A_r)(P_b)}{(V_b - V_r)(P_b - P_v)} \qquad \text{Eq. 10A-1}$$

NOTE: A and A_r must be at the same wavelength.

12.3 CO Concentration in the Bag. Calculate C_b using Equations 10A–2 and 10A–3. If condensate is visible in the bag, calculate B_w using Table 10A–2 and the temperature and barometric pressure in the analysis room. If condensate is not visible, calculate B_w using the temperature and barometric pressure at the sampling site.

$$B_w = \frac{P_w}{P_b} \qquad \text{Eq. 10A-2}$$

$$C_b = \frac{C_g}{(1 - B_w)} \qquad \text{Eq. 10A-3}$$

12.4 CO Concentration in the Stack.

$$C = C_b\ (1 - F) \qquad \text{Eq. 10A-4}$$

13.0 Method Performance

13.1 Precision. The estimated intralaboratory standard deviation of the method is 3 percent of the mean for gas samples analyzed in duplicate in the concentration range of 39 to 412 ppm. The interlaboratory precision has not been established.

13.2 Accuracy. The method contains no significant biases when compared to an NDIR analyzer calibrated with NIST standards.

13.3 Range. Approximately 3 to 1800 ppm CO. Samples having concentrations below 400 ppm are analyzed at 425 nm, and samples having concentrations above 400 ppm are analyzed at 600 nm.

13.4 Sensitivity. The detection limit is 3 ppmv based on a change in concentration equal to three times the standard deviation of the reagent blank solution.

13.5 Stability. The individual components of the colorimetric reagent are stable for at least one month. The colorimetric reagent must be used within two days after preparation to avoid excessive blank correction. The samples in the bag should be stable for at least one week if the bags are leak-free.

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

1. Butler, F.E., J.E. Knoll, and M.R. Midgett. Development and Evaluation of Methods for Determining Carbon Monoxide Emissions. U.S. Environmental Protection Agency, Research Triangle Park, N.C. June 1985. 33 pp.

2. Ferguson, B.B., R.E. Lester, and W.J. Mitchell. Field Evaluation of Carbon Monoxide and Hydrogen Sulfide Continuous Emission Monitors at an Oil Refinery. U.S. Environmental Protection Agency, Research Triangle Park, N.C. Publication No. EPA–600/4–82–054. August 1982. 100 pp.

3. Lambert, J.L., and R.E. Weins. Induced Colorimetric Method for Carbon Monoxide. Analytical Chemistry. *46*(7):929–930. June 1974.

4. Levaggi, D.A., and M. Feldstein. The Colorimetric Determination of Low Concentrations of Carbon Monoxide. Industrial Hygiene Journal. *25*:64–66. January-February 1964.

5. Repp, M. Evaluation of Continuous Monitors For Carbon Monoxide in Stationary Sources. U.S. Environmental Protection Agency, Research Triangle Park, N.C. Publication No. EPA–600/2–77–063. March 1977. 155 pp.

6. Smith, F., D.E. Wagoner, and R.P. Donovan. Guidelines for Development of a Quality Assurance Program: Volume VIII—Determination of CO Emissions from Stationary Sources by NDIR Spectrometry. U.S. Environmental Protection Agency, Research Triangle Park, N.C. Publication No. EPA–650/4–74–005–h. February 1975. 96 pp.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 10A–1—DATA RECORDING SHEET FOR SAMPLES ANALYZED IN TRIPLICATE

Sample No./type	Room temp °C	Stack %CO_2	Bulb No.	Bulb vol. liters	Reagent vol. in bulb, liter	Partial pressure of gas in bulb, mm Hg	P_b, mm Hg	Shaking time, min	Abs versus water	A-A_r	A_s	Avg A_s
blank												
Std. 1												
Std. 2												
Sample 1												
Sample 2												
Sample 3												

TABLE 10A–2—MOISTURE CORRECTION

Temperature °C	Vapor pressure of H_2O, mm Hg	Temperature °C	Vapor pressure of H_2, mm Hg
4	6.1	18	15.5
6	7.0	20	17.5
8	8.0	22	19.8
10	9.2	24	22.4
12	10.5	26	25.2
14	12.0	28	28.3
16	13.6	30	31.8

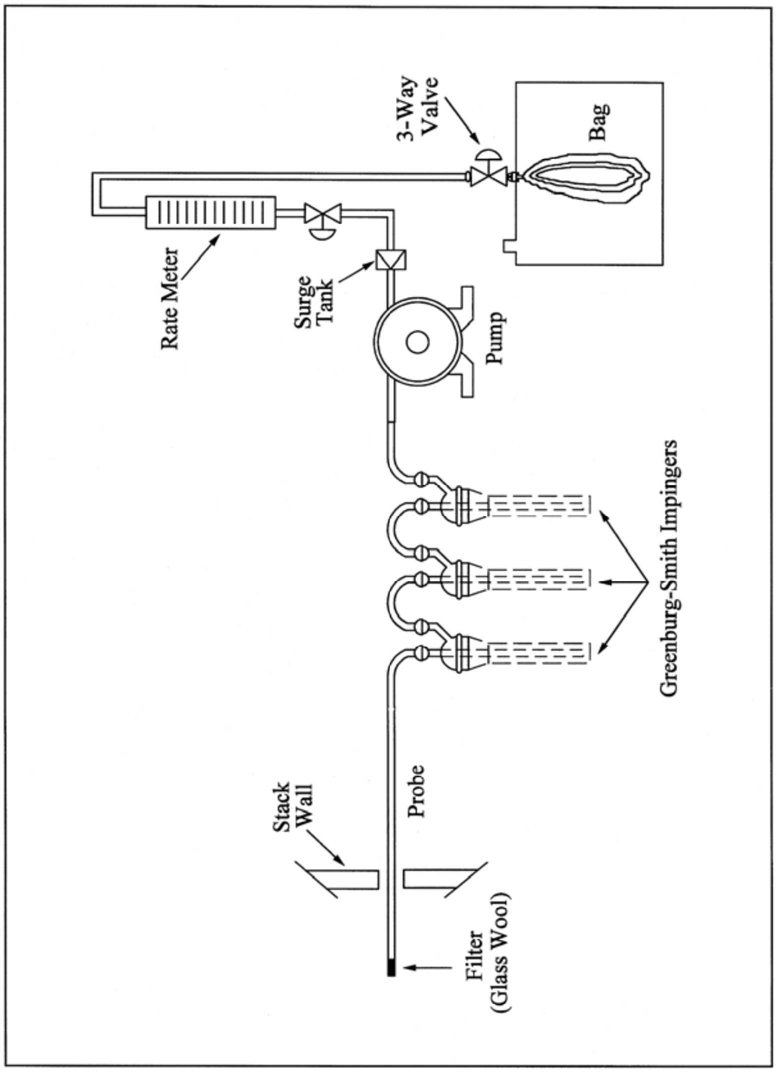


Figure 10A-1. Sampling Train.

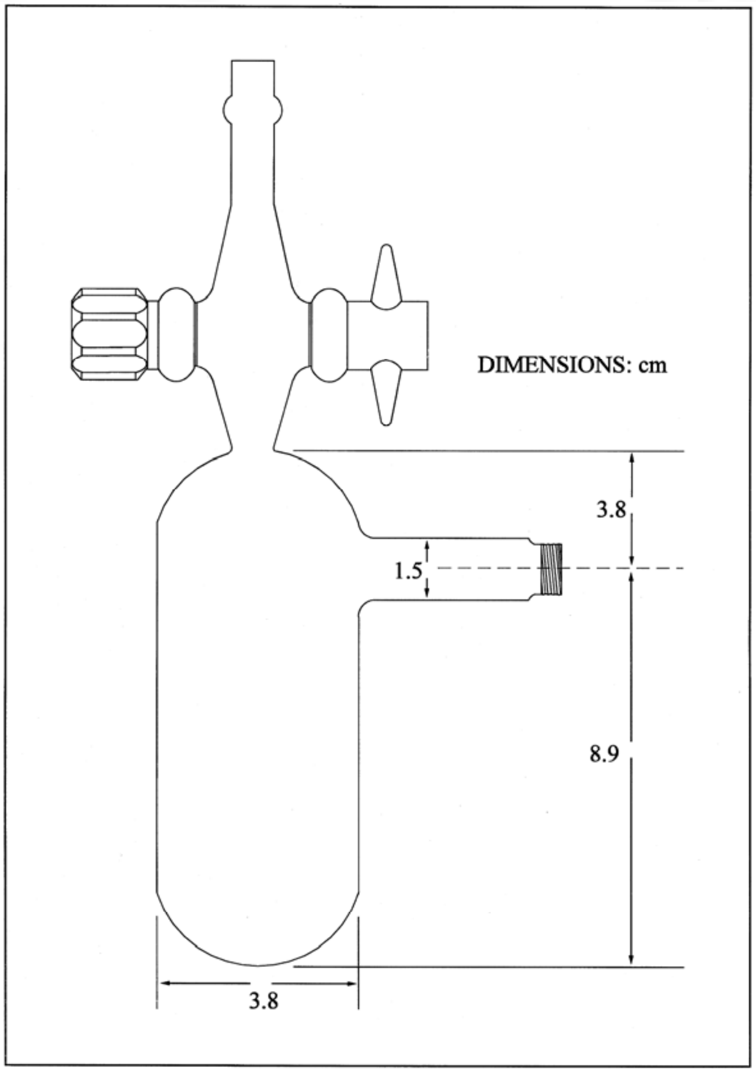


Figure 10A-2. Sample Reaction Bulbs.

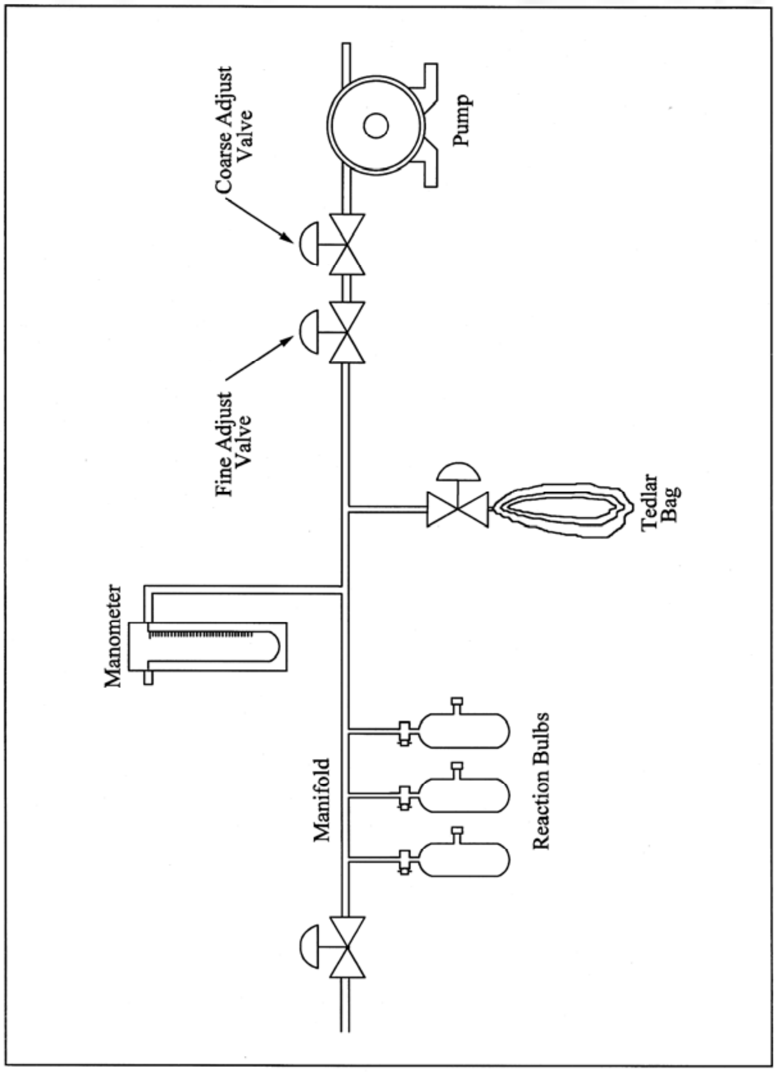


Figure 10A-3. Sample Bulb Filling System.

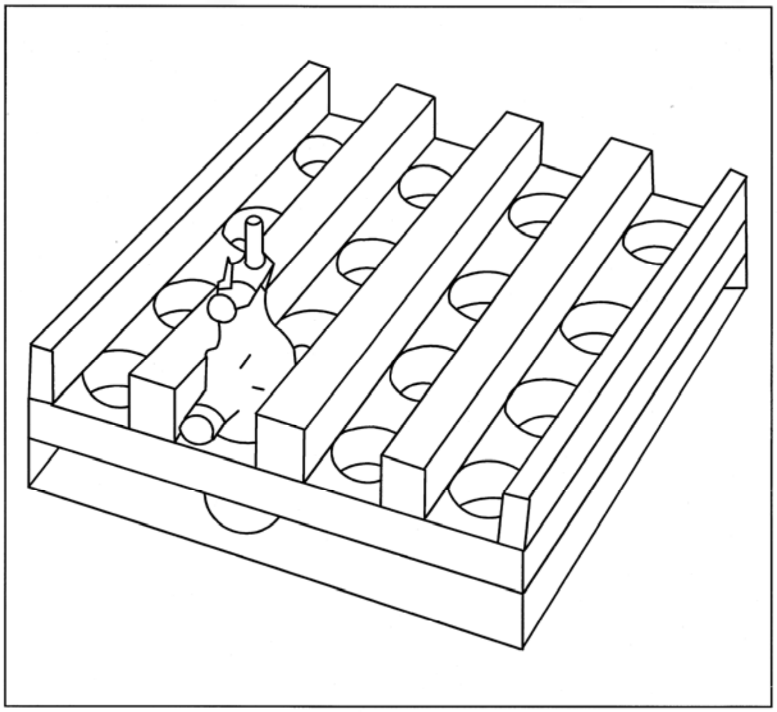


Figure 10A-4. Shaker Table Adapter.

METHOD 10B—DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM STATIONARY SOURCES

NOTE: This method is not inclusive with respect to specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 4, Method 10A, and Method 25.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Carbon monoxide (CO)	630–08–0	Not determined.

1.2 Applicability. This method applies to the measurement of CO emissions at petroleum refineries and from other sources when specified in an applicable subpart of the regulations.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 An integrated gas sample is extracted from the sampling point, passed through a conditioning system to remove interferences, and collected in a Tedlar or equivalent bag. (Verify through the manufacturer that the Tedlar alternative is suitable for NO and make this verifying information available for inspection.) The CO is separated from the sample by gas chromatography (GC) and catalytically reduced to methane (CH_4) which is determined by flame ionization detection (FID). The analytical portion of this method is identical to applicable sections in Method 25 detailing CO measurement.

3.0 Definitions [Reserved]

4.0 Interferences

4.1 Carbon dioxide (CO_2) and organics potentially can interfere with the analysis. Most of the CO_2 is removed from the sample by the alkaline permanganate conditioning system; any residual CO_2 and organics are separated from the CO by GC.

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method. The analyzer users manual should be consulted for specific precautions concerning the analytical procedure.

6.0 Equipment and Supplies

6.1 Sample Collection. Same as in Method 10A, section 6.1 (paragraphs 6.1.1 through 6.1.11).

6.2 Sample Analysis. A GC/FID analyzer, capable of quantifying CO in the sample and consisting of at least the following major components, is required for sample analysis. [Alternatively, complete Method 25 analytical systems (Method 25, section 6.3) are acceptable alternatives when calibrated for CO and operated in accordance with the Method 25 analytical procedures (Method 25, section 11.0).]

6.2.1 Separation Column. A column capable of separating CO from CO_2 and organic compounds that may be present. A 3.2-mm (⅛-in.) OD stainless steel column packed with 1.7 m (5.5 ft.) of 60/80 mesh Carbosieve S-II (available from Supelco) has been used successfully for this purpose.

6.2.2 Reduction Catalyst. Same as in Method 25, section 6.3.1.2.

6.2.3 Sample Injection System. Same as in Method 25, Section 6.3.1.4, equipped to accept a sample line from the bag.

6.2.4 Flame Ionization Detector. Meeting the linearity specifications of section 10.3 and having a minimal instrument range of 10 to 1,000 ppm CO.

6.2.5 Data Recording System. Analog strip chart recorder or digital integration system, compatible with the FID, for permanently recording the analytical results.

7.0 Reagents and Standards

7.1 Sample Collection. Same as in Method 10A, section 7.1.

7.2 Sample Analysis.

7.2.1 Carrier, Fuel, and Combustion Gases. Same as in Method 25, sections 7.2.1, 7.2.2, and 7.2.3, respectively.

7.2.2 Calibration Gases. Three standard gases with nominal CO concentrations of 20, 200, and 1,000 ppm CO in nitrogen. The calibration gases shall be certified by the manufacturer to be ±2 percent of the specified concentrations.

7.2.3 Reduction Catalyst Efficiency Check Calibration Gas. Standard CH_4 gas with a nominal concentration of 1,000 ppm in air.

8.0 Sample Collection, Preservation, Storage, and Transport

Same as in Method 10A, section 8.0.

9.0 Quality Control

Section	Quality control measure	Effect
8.0	Sample bag/sampling system leak-checks	Ensures that negative bias introduced through leakage is minimized.
10.1	Carrier gas blank check	Ensures that positive bias introduced by contamination of carrier gas is less than 5 ppmv.
10.2	Reduction catalyst efficiency check	Ensures that negative bias introduced by inefficient reduction catalyst is less than 5 percent.
10.3	Analyzer calibration	Ensures linearity of analyzer response to standards.
11.2	Triplicate sample analyses	Ensures precision of analytical results.

10.0 Calibration and Standardization

10.1 Carrier Gas Blank Check. Analyze each new tank of carrier gas with the GC analyzer according to section 11.2 to check for contamination. The corresponding concentration must be less than 5 ppm for the tank to be acceptable for use.

10.2 Reduction Catalyst Efficiency Check. Prior to initial use, the reduction catalyst shall be tested for reduction efficiency. With the heated reduction catalyst bypassed, make triplicate injections of the 1,000 ppm CH_4 gas (Section 7.2.3) to calibrate the analyzer. Repeat the procedure using 1,000 ppm CO gas (Section 7.2.2) with the catalyst in operation. The reduction catalyst operation is acceptable if the CO response is within 5 percent of the certified gas value.

10.3 Analyzer Calibration. Perform this test before the system is first placed into operation. With the reduction catalyst in operation, conduct a linearity check of the analyzer using the standards specified in section 7.2.2. Make triplicate injections of each calibration gas, and then calculate the average response factor (area/ppm) for each gas, as well as the overall mean of the response factor values. The instrument linearity is acceptable if the average response factor of each calibration gas is within 2.5 percent of the overall mean value and if the relative standard deviation (calculated in section 12.8 of Method 25) for each set of triplicate injections is less than 2 percent. Record the overall mean of the response factor values as the calibration response factor (R).

11.0 Analytical Procedure

11.1 Preparation for Analysis. Before putting the GC analyzer into routine operation, conduct the calibration procedures listed in section 10.0. Establish an appropriate carrier flow rate and detector temperature for the specific instrument used.

11.2 Sample Analysis. Purge the sample loop with sample, and then inject the sample. Analyze each sample in triplicate, and calculate the average sample area (A). Determine the bag CO concentration according to section 12.2.

12.0 Calculations and Data Analysis

Carry out calculations retaining at least one extra significant figure beyond that of the acquired data. Round off results only after the final calculation.

12.1 Nomenclature.

A = Average sample area.
B_w = Moisture content in the bag sample, fraction.
C = CO concentration in the stack gas, dry basis, ppm.
C_b = CO concentration in the bag sample, dry basis, ppm.
F = Volume fraction of CO_2 in the stack, fraction.
P_{bar} = Barometric pressure, mm Hg.
P_w = Vapor pressure of the H_2O in the bag (from Table 10A–2, Method 10A), mm Hg.
R = Mean calibration response factor, area/ppm.

12.2 CO Concentration in the Bag. Calculate C_b using Equations 10B–1 and 10B–2. If condensate is visible in the bag, calculate B_w using Table 10A–2 of Method 10A and the temperature and barometric pressure in the analysis room. If condensate is not visible, calculate B_w using the temperature and barometric pressure at the sampling site.

$$B_w = \frac{P_w}{P_{bar}} \qquad \text{Eq. 10B-1}$$

$$C_b = \frac{A}{R\left(1 - B_w\right)} \qquad \text{Eq. 10B-2}$$

12.3 CO Concentration in the Stack

$$C = C_b\,(1 - F) \qquad \text{Eq. 10B-3}$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as in Method 25, section 16.0, with the addition of the following:

1. Butler, F.E., J.E. Knoll, and M.R. Midgett. Development and Evaluation of Methods for Determining Carbon Monoxide Emissions. Quality Assurance Division, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC. June 1985. 33 pp.

17.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

[36 FR 24877, Dec. 23, 1971]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting appendix A–4 to part 60, see the List of CFR sections Affected, which appears in the Finding Aids section of the printed volume and at *www.fdsys.gov.*

APPENDIX A–5 TO PART 60—TEST METHODS 11 THROUGH 15A

Method 11—Determination of hydrogen sulfide content of fuel gas streams in petroleum refineries
Method 12—Determination of inorganic lead emissions from stationary sources
Method 13A—Determination of total fluoride emissions from stationary sources—SPADNS zirconium lake method
Method 13B—Determination of total fluoride emissions from stationary sources—Specific ion electrode method
Method 14—Determination of fluoride emissions from potroom roof monitors for primary aluminum plants
Method 14A—Determination of Total Fluoride Emissions from Selected Sources at Primary Aluminum Production Facilities
Method 15—Determination of hydrogen sulfide, carbonyl sulfide, and carbon disulfide emissions from stationary sources
Method 15A—Determination of total reduced sulfur emissions from sulfur recovery plants in petroleum refineries

The test methods in this appendix are referred to in §60.8 (Performance Tests) and §60.11 (Compliance With Standards and Maintenance Requirements) of 40 CFR part 60, subpart A (General Provisions). Specific uses of these test methods are described in the standards of performance contained in the subparts, beginning with Subpart D.

Within each standard of performance, a section title "Test Methods and Procedures" is provided to: (1) Identify the test methods