

Section 3.8

METHOD 10-DETERMINATION OF CARBON MONOXIDE EMISSIONS FROM  
STATIONARY SOURCES

OUTLINE

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### SUMMARY

A gas sample is extracted from the stack either at a constant rate using a continuous sampling train or at a rate proportional to the stack velocity using an integrated sampling train. The concentration of carbon monoxide (CO) from both sampling methods is determined by a Luft-type nondispersive infrared (NDIR) analyzer or equivalent analyzer. The method is applicable to stationary sources when specified by a compliance procedure and/or when the CO concentration is  $\geq 20$  parts per million (ppm) for a 0-to-1000-ppm testing range. With this method, interferences can result from substances with strong infrared absorption energies. Interference ratios in the 1500-to-3000-ppm testing range are 7 ppm CO per 3.5% for water (H<sub>2</sub>O) and 10 ppm CO per 10% for carbon dioxide (CO<sub>2</sub>). In the 0-to-100-ppm range, they can be as high as 25 ppm CO per 3.5% H<sub>2</sub>O and 50 ppm CO per 10% CO<sub>2</sub>. Major interferences can be avoided by using silica gel and ascarite traps to remove H<sub>2</sub>O and CO<sub>2</sub>, respectively; if traps are used, the gas sample volumes must be adjusted. The method description given herein is based on the Reference Method promulgated March 8, 1974 (Section 3.8.10) and on collaborative testing.<sup>1</sup> Blank forms for recording data are provided in the Method Highlights and in Section 3.8.12 for the convenience of Handbook users.

#### METHOD HIGHLIGHTS

Section 3.8 describes specifications for determination of carbon monoxide (CO) from stationary sources. A gas sample is extracted from the stack using a continuous or integrated sampling train and analyzed using a Luft-type nondispersive infrared (NDIR) analyzer or the equivalent. Interferences include any substance having a strong absorption of infrared energy. Major interference problems caused by water (H<sub>2</sub>O) and carbon dioxide (CO<sub>2</sub>) are removed using silica gel and ascarite traps, respectively.

Continuous sampling is performed by connecting the NDIR to the continuous sampling train and conducting the analysis. Integrated sampling is performed by withdrawing a sample at a rate proportional to stack gas velocity, into a Tedlar, or equivalent evacuated bag. CO<sub>2</sub> content, for each sampling method can be determined using the Method 3 integrated sampling procedure or by weighing the ascarite CO<sub>2</sub> removal trap and computing CO<sub>2</sub> concentration from the gas volume sampled and the weight gain of the trap. Results of collaborative tests<sup>1,2</sup> of Method 10 revealed several problems which, if eliminated, may result in improved precision and accuracy. Reference gases were cited as an area where improved quality control is needed. A need for further training of NDIR operators was also cited as another area that needed improvement.

The blank data forms at the end of the highlights section may be removed from the Handbook and used in the pretest, test, and posttest operations. Each form has a subtitle (e.g., Method 10, Figure 5.1) to assist the user in finding a similar filled-in form in the Method Description (e.g., in Section 3.8.5). On the blank and filled-in forms, the item/parameters that can cause the most significant errors are indicated with an asterisk.

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1. Procurement of Equipment

Section 3.8.1 (Procurement of Apparatus and Supplies) gives the specifications, criteria, and design features for equipment and materials required for performing Method 10 tests. This section is designed to serve as a guide in the procurement and initial check of equipment and supplies. The activity matrix (Table 1.1) at the end of Section 3.8.1 can be used as a quick reference; it follows the same order as the written description in the main text.

2. Pretest Preparations

Section 3.8.2 (Calibration of Apparatus) provides a step-by-step description of the required calibration procedures. Detailed methods and procedures are described for calibrating the NDIR. The calibration section can be removed and compiled, along with calibration sections from all other methods, into a separate quality assurance reference manual for use by calibration personnel. A pretest checklist (Figure 3.1) or similar form should be used to summarize the calibration data.

Section 3.8.3 (Presampling Operations) provides the tester with a guide for supplies and equipment preparation for field tests. The pretest preparation form (Figure 3.2) can be used as an equipment checkout and packing list. The method for packing and the recommended packing containers should help protect the equipment, but are not required.

3. On-Site Measurements

Section 3.8.4 (On-Site Measurements) contains step-by-step procedures for sampling using the continuous and integrated methods. The procedure for continuous sampling includes performing the analysis on-site at the time of sample collection; therefore, procedures for analyzing the continuous sample are included in this section. The on-site measurement checklist (Figure 4.4) is provided to assist the tester with a quick method of checking requirements.

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#### 4. Posttest Operations

Section 3.8.5 (Postsampling Operations) gives the posttest equipment check procedures and a step-by-step analytical procedure for integrated samples. Figure 5.1 or a similar form should be used to summarize the posttest calibration checks and should be included in the emission test report.

Section 3.8.6 (Calculations) provides the tester with the required equations, nomenclature, and suggested number of significant digits. It is suggested that, if available, a programmable calculator be used to reduce chance of calculation error.

Section 3.8.7 (Maintenance) supplies the tester with a guide for a routine maintenance program. The program is not a requirement, but is suggested for reducing equipment malfunctions.

#### 5. Auditing Procedure

Section 3.8.8 (Auditing Procedure) provides a description of necessary activities for conducting performance and system audits. The performance audits include an audit of the analytical phase and an audit of data processing. A system audit consists of an on-site qualitative evaluation of the test team performance. The performance and system audits provide an independent assessment of data quality.

Section 3.8.9 (Recommended Standards for Establishing Traceability) recommends the primary standards to which the sample collection and analysis should be traceable.

#### 6. References

Section 3.8.10 (Reference Method) contains a copy of the EPA Reference Method.

Section 3.8.11 (References) provides the reader with a list of all the references used in the compilation of this section of the Handbook along with additional sources.

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PRETEST SAMPLING CHECKS  
(Method 10, Figure 3.1)

Date \_\_\_\_\_ Completed by \_\_\_\_\_

Pitot Tube

Identification number \_\_\_\_\_ Date \_\_\_\_\_

Dimensional specifications checked?\* \_\_\_\_\_ yes \_\_\_\_\_ no

Calibration required? \_\_\_\_\_ yes \_\_\_\_\_ no

Date \_\_\_\_\_  $C_p$  \_\_\_\_\_

Rotameter

Identification number \_\_\_\_\_

Calibration required?\* \_\_\_\_\_ yes \_\_\_\_\_ no

Barometer

Calibrated?\* \_\_\_\_\_ yes \_\_\_\_\_ no

\*Most significant items/parameters to be checked.

PRETEST PREPARATIONS  
 (Method 10, Figure 3.2)

Apparatus check	Acceptable		Quantity required	Ready		Loaded and packed
	Yes	No		Yes	No	
<u>Probe</u>						
Pyrex glass _____ Stainless steel _____						
<u>Filter</u> _____						
<u>Pitot tube</u>						
Type _____ Length _____ Calibrated* _____ Differential pressure gauge _____						
<u>Air-cooled condenser</u>						
Clean _____ Leak checked* _____						
<u>Needle valve and rotameter</u>						
Clean _____ Calibrated* _____						
<u>Barometer</u>						
Type _____ Calibrated* _____						
<u>Pump</u>						
Type _____ Leak checked _____						
<u>Flexible bag</u>						
Type _____ Leak checked* _____ Evacuated* _____						

\*Most significant items/parameters to be checked.

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ON-SITE MEASUREMENTS CHECKLIST  
(Method 10, Figure 4.4)

Continuous Sampling

Leak check prior to sampling (optional) \_\_\_\_\_  
NDIR analyzer allowed to warm up (1 h minimum)\* \_\_\_\_\_  
Multipoint calibration curve constructed\* \_\_\_\_\_  
Sampling port plugged \_\_\_\_\_  
Sampling flow rate properly set (manufacturer's recommended and  
<1 l/min)\* \_\_\_\_\_  
Sampling system properly purged\* \_\_\_\_\_  
Posttest leak check (mandatory)\* \_\_\_\_\_  
All data properly recorded\* \_\_\_\_\_  
CO<sub>2</sub> concentration determined\* \_\_\_\_\_

Integrated Sampling

Sampling rate selected for integrated sampling \_\_\_\_\_  
Leak check prior to sampling (optional) \_\_\_\_\_  
Sampling port plugged \_\_\_\_\_  
Sampling train purged (5 times system volume or 10 min)\* \_\_\_\_\_  
Flexible bag properly sealed and labeled\* \_\_\_\_\_  
Posttest leak check (mandatory)\* \_\_\_\_\_  
All data properly recorded\* \_\_\_\_\_  
CO<sub>2</sub> concentration determined\* \_\_\_\_\_

\*Most significant item/parameters to be checked.

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POSTTEST SAMPLING CHECKS  
(Method 10, Figure 5.1)

NDIR

Posttest zero check \_\_\_\_\_ adjusted value \_\_\_\_\_  
Posttest span check\* \_\_\_\_\_ within  $\pm 10\%$  of pretest calibration  
Recalibration required? \_\_\_\_\_ yes \_\_\_\_\_ no  
If yes, void all data back to the last calibration check that  
was within the  $\pm 10\%$  limit

Rotameter

Pretest calibration factor,  $Y_r$  \_\_\_\_\_ within  $\pm 5\%$   
Posttest check, \*  $Y_r$  \_\_\_\_\_ within  $\pm 10\%$  of pretest  
Recalibration recommended? \_\_\_\_\_ yes \_\_\_\_\_ no  
If performed, recalibration factor,  $Y_r$  \_\_\_\_\_ ( $Y_r$  not used for  
emission calculations)  
Rotameter cleaned? \_\_\_\_\_ yes \_\_\_\_\_ no

Analysis (Integrated Samples)

Calibration gases traceable to NBS standard gas\* \_\_\_\_\_  
NDIR allowed to warm up (1 h minimum)\* \_\_\_\_\_  
Multipoint calibration curve constructed\* \_\_\_\_\_  
Sampling lines and analyzer properly purged (5 times system  
volume or 10 min)\* \_\_\_\_\_  
Three successive readings made from each bag \_\_\_\_\_  
Highest and lowest values differ by  $\leq 5\%$  \_\_\_\_\_

\*Most significant items/parameters to be checked.

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## 1.0 PROCUREMENT OF APPARATUS AND SUPPLIES

Carbon monoxide (CO) sampling trains used to obtain integrated and continuous gas samples are shown in Figures 1.1 and 1.2, respectively. Table 1.1 at the end of this section summarizes the quality assurance activities for procurement and acceptance of apparatus and supplies.

Specifications, criteria, and/or design features as applicable are given in this section to aid in the selection of equipment to ensure the collection of good quality data. Procedures and limits for acceptance checks, where applicable, are given.

During the procurement of equipment and supplies, it is suggested that a procurement log (Figure 1.3) be used to record the descriptive title of the equipment; the identification number, if applicable; and the results of the acceptance checks. Also, if calibration is required as part of the acceptance check, the data are to be recorded in the calibration log book.

### 1.1 Sampling

1.1.1 Sampling Probe - The sampling probe should consist of a 316 seamless stainless steel tube or a sheathed borosilicate (Pyrex) glass tube with an inside diameter (ID) of approximately 6 mm (0.24 in.); and equipped with an in-stack or out-stack particulate filter. When an in-stack filter is used, the probe should have an expanded ID of 38 to 40 mm (1.5 to 1.6 in.) for the first 40 mm (1.6 in.) of the probe inlet. The expanded section should be packed with glass wool prior to sampling. The probe outlet must have a fitting suitable for attachment to an air-cooled condenser inlet. A probe approximately 1.1 m (4 ft) long is usually sufficient; the exact length can be determined after a sampling site inspection. If the stack must be traversed to obtain an integrated sample, the probe length should be chosen accordingly.

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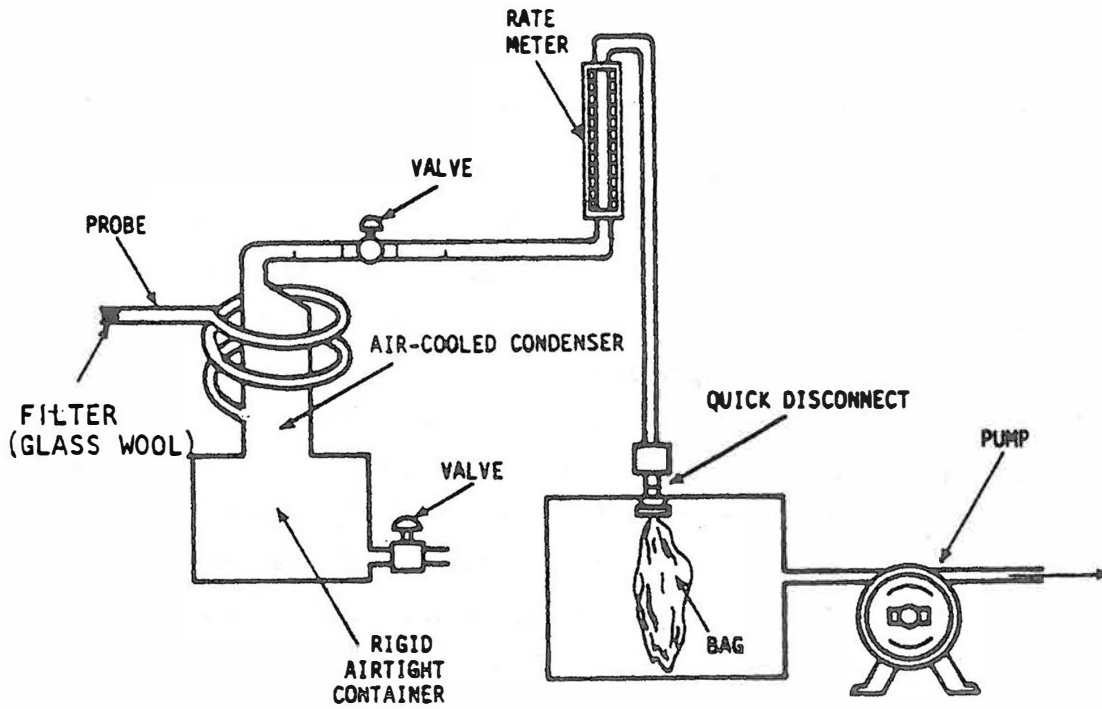


Figure 1.1. Sampling train for integrated analysis.

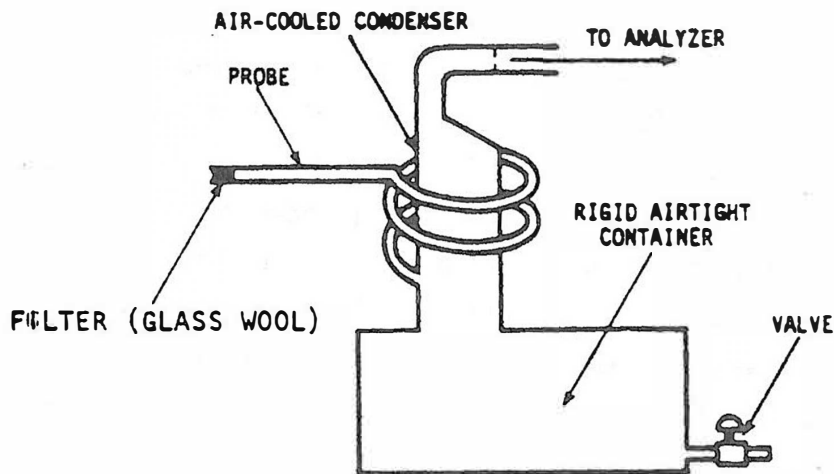


Figure 1.2. Sampling train for continuous analysis.

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			
CONDENSER	3	17764014	ACE METAL Co	2-5-78	3-8-78	\$ 250	READY FOR USE	IRC

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Figure 1.3. Example of a procurement log.

The main criterion in selecting the probe material is that it be nonreactive with the gas constituents so that it will not introduce bias into the analysis.

Upon receiving a new probe, visually check it for adherence to specifications (i.e., the length and composition ordered). Check for breaks, cracks, and leaks. Leak check the probe; connect it to a pump inlet, plug other end, and pull a 380 mm (15 in.) Hg vacuum. Leakage rates  $>0.00057 \text{ m}^3/\text{min}$  ( $0.02 \text{ ft}^3/\text{min}$ ) measured by the dry gas meter are unacceptable. Any probe not satisfying the checks should be repaired if possible or returned to the supplier.

1.1.2 Air-Cooled Condenser - The condenser facilitates the condensation of water from the gas being sampled. The coiled tubes (Figures 1.1 and 1.2) allow the entering gas to cool to near ambient temperature; lower temperatures can be obtained by using a circulating water cooler or an ice bath.

The reservoir collects and holds the condensed water until it is drained between sampling runs by a valve. The capacity of the reservoir must be sufficient to collect all condensed moisture from the gas during system purging and sampling, but it should not be unnecessarily oversized because the added size would increase the bulk of the sampling train and lengthen purging time. For example, a sampling train of 1- $\ell$  volume (including the condenser) should hold the condensate from about 100  $\ell$  of gas (90- $\ell$  sample plus 5- $\ell$  displacements of the sampling train volume plus 5- $\ell$  margin). With 20% water concentration in the stack gases, the 100- $\ell$  sample would contain 20  $\ell$  of water vapor; when condensed, the 20  $\ell$  of vapor would correspond to about 20 ml of water; therefore, a condenser volume of 0.25  $\ell$  would allow an adequate operating margin. The amount of water that would be collected can be estimated either from knowledge of the process or by determining the moisture content (Method 3) and sample volume.

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Check the condenser visually for damage, breaks, cracks, and manufacturing flaws. The condenser should be leak free at a draft gauge positive pressure of 25 mm (1 in. H<sub>2</sub>O) and at a vacuum gauge reading of 380 mm (15 in.) Hg. If the condenser is defective, repair it or return it to the supplier.

1.1.3 Needle Valve - A stainless steel needle valve with appropriate fittings to make it leak free is recommended to regulate the flow in the sampling train. Install the valve and check for proper operation. If the valve is defective or if it cannot regulate the sample flow over the 0-to-1 l/min (0-to-0.035 ft<sup>3</sup>/min) range, repair it or return it to the supplier.

1.1.4 Vacuum Pump - The vacuum pump should be capable of maintaining a flow rate of 1 l/min (0.035 ft<sup>3</sup>/min) at 380 mm (15 in.) Hg. A leak-free diaphragm pump (or the equivalent) must be used because of inherently low contamination possibilities with this type of pump.

A new pump should be visually checked for damage, leaks, and capacity upon receipt. To leak check the pump, install a vacuum gauge in the pump inlet line; plug the inlet line, and run the pump until the vacuum gauge reads 380 mm (15 in.) Hg; then close the pump outlet line, and turn the pump off. The vacuum gauge should remain stable for 30 s. If defective, return it to the supplier.

1.1.5 Rate Meter - The rate meter is a rotameter (or equivalent) used to measure the sample gas flow rate in the range of 0 - 1 l/min (0-to-0.035 ft<sup>3</sup>/min). Inspect the rotameter for cracks, flaws, and erratic behavior, and check calibration as described in Section 3.8.2. Return it to the supplier if it is damaged or cannot be adjusted to within ±5% of the standard rate meter. Clean and recalibrate if dust and/or liquid contamination is suspected.

1.1.6 Flexible Bag - The flexible bag used to obtain the integrated gas sample should be leak free and made of Tedlar (or an equivalent material) with a capacity of 60-to-90 l (2 to 3 ft<sup>3</sup>).

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Upon receipt of a new bag, check for damage, correct fittings, and capacity. Before using, leak check in the laboratory by evacuating the bag with a leakless pump. When evacuation is complete, there should be no flow through the dry gas meter. In leak testing by evacuation, it is difficult to ascertain whether the entire bag has been tested. If one wall of the bag presses against another section and eventually cuts off the flow, the absence of flow does not guarantee that all sections of the bag are leak free. Therefore, an alternative and preferred test is to pressurize the bag with air to approximately 51 mm (2 in.) H<sub>2</sub>O above atmospheric pressure and to monitor the pressure with a draft gauge over a period of time. Loss of pressure over a 24-h period should be considered an excessive leak, and the bag should be repaired or replaced.

1.1.7 Pitot Tube - The pitot tube should be a Type S (or equivalent) as described in Section 3.1 of this Handbook. The pitot tube is to be used when the sampling rate is regulated proportionally to the stack gas velocity (integrated sample), when the velocity is varying over time, or when a velocity traverse (flow rate determination) is conducted.

1.1.8 Wet Test Meter - The wet test meter is used to check the calibration of the rotameter. The wet test meter should be capable of measuring a volume of 2 l (0.070 ft<sup>3</sup>) with an accuracy of  $\pm 1\%$  at a flow rate of 1 l/min (0.035 ft<sup>3</sup>/min).

Upon receiving a wet test meter, visually check it for manufacturing defects and leaks, and calibrate it as described in Section 3.8.2. If it is damaged, behaves erratically, or cannot be properly adjusted, return it to the manufacturer.

1.1.9 Barometer - A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg may be used; however, in many cases the absolute barometric pressure can be obtained from a nearby weather service station. If the elevation of the sampling point is higher than that of the weather station the reported barometric pressure is reduced at a

rate of 2.5 mm Hg/30 m (0.1 in. Hg/100 ft) of elevation difference; if the sampling point is lower than the weather station, the pressure is increased at the same rate. Note: Make sure the weather service station gives the pressure without correction to sea level.

Check the field barometer against a mercury-in-glass barometer (or its equivalent). If the field barometer cannot be adjusted to agree with the mercury-in-glass barometer, it is not acceptable.

1.1.10 Vacuum Gauge - A vacuum gauge capable of measuring at least 760 mm (30 in.) Hg is to be used to leak check the sampling train. Check the vacuum gauge in a parallel leakless system with a mercury U-tube manometer at a vacuum of 380 mm (15 in.) Hg. Be sure the gauge agrees within  $\pm 25$  mm (1.0 in.) Hg. If it does not, adjust or reject.

## 1.2 Sample Analysis

1.2.1 Carbon Monoxide Analyzer - The CO analyzer should be a Luft-type nondispersive infrared (NDIR) spectrometer (or equivalent), which meets or exceeds the specifications in Appendices A and B in Section 3.8.10. When purchasing a CO analyzer, have the manufacturer demonstrate that it meets these specifications as well as those advertised by the manufacturer. The best evidence is a strip chart record of that analyzer's performance. Guidelines for instrument evaluation are given in "Procedures for Testing Performance Characteristics of Automated Methods," Federal Register, Vol. 40, No. 33, February 18, 1975. If the instrument is defective, return it to the manufacturer for repair, adjustment, or replacement.

1.2.2 Drying Tube - A drying tube packed with 6 to 16 mesh indicating-type silica gel (or equivalent) should be installed to remove moisture from the sample. If not removed, the moisture may interfere with the NDIR measurement of CO.

The tube can be made of stainless steel, glass, or plastic. Each end of the tube should be packed with glass wool to prevent the silica gel from entering the sampling train and the NDIR

analyzer. The tube should be leak free at a vacuum of at least 380 mm (15 in.) Hg, and it should have a minimum capacity of 200 g of silica gel. If defective, repair or return to the supplier.

1.2.3 Carbon Dioxide Removal Tube - Install a flexible plastic tube packed with 500 g of ascarite to remove CO<sub>2</sub> from the sample. If not removed, the CO<sub>2</sub> may interfere with the NDIR measurement of CO. Keep tube in a vertical orientation to prevent channeling.

A flexible plastic tube with a minimum ID of 2.5 cm (1.0 in.) and capped at the ends should be used (instead of a rigid container). When CO<sub>2</sub> contacts the ascarite, it tends to form a dense solid plug which can easily block the glass inlet tube of an impinger. The inlet and outlet lines of the tube should be configured to maximize exposure of sample gas to the ascarite and to prevent plugging. Pack each end of the tube with glass wool to protect the sampling train and the NDIR analyzer from ascarite dust. Inspect the CO<sub>2</sub> removal tube for breaks, damage, and correct fittings; it should be leak free at a vacuum of 380 mm (15 in.) Hg. If defective, return to the supplier.

The drying tube and the CO<sub>2</sub> removal tube may be combined into one unit containing layers of the two materials in the quantities previously noted. The sample should pass first into pure silica gel, then into a layer of silica gel and ascarite, and finally through a layer of ascarite. Repack this tube with silica gel and ascarite when the existing silica gel exhibits the characteristic color change.

1.2.4 Filter - Place a filter in the sample in-take line of the CO analyzer to remove particulates from the gas stream and to prevent erroneous results and damage to the NDIR analyzer. If the manufacturer of the CO analyzer specifies a filter type and size, those should be followed; if not, a standard glass fiber filter (e.g., MSA1106BH or the equivalent) can be used.

Upon receiving the filter, check for specifications (i.e., the type and size ordered). If incorrect, return to the manufacturer.

1.2.5 Ice Water Bath - The drying tube and the CO<sub>2</sub> removal tube should be watertight and should be immersed in an ice water bath of sufficient size to contain and ensure efficient operation of the tubes. If the size is incorrect, return the bath to the supplier.

1.2.6 Carbon Dioxide Analyzer - Carbon dioxide in the gas stream interferes with NDIR readings, thus use an ascarite trap to remove all CO<sub>2</sub>. To correct the CO reading for the removed CO<sub>2</sub>, the percentage of CO<sub>2</sub> in the stream must be determined by using an Orsat analyzer, as specified in Section 3.2 of the Handbook.

1.2.7 Recorder - A strip chart recorder is optional; however, it provides a permanent record of NDIR readings. When ordering a recorder, make sure that its operating voltage is compatible with the NDIR voltage output.

Upon receiving a recorder, check for damage and proper operation throughout its entire input voltage range. If defective, return it to the supplier.

### 1.3 Reagents

1.3.1 Calibration Gases - A multipoint calibration for the selected measurement range of the CO analyzer requires three known concentrations of calibration gases: one concentration of CO in nitrogen (N<sub>2</sub>) for the upper value (span) of the selected range and two CO concentrations at 30% and 60% of span. In addition, a prepurified grade of N<sub>2</sub> (containing <0.1 ppm CO) is required for a zero gas.

The analyzer range cannot exceed the source performance standard by >1½ times. For example, the standard for petroleum refineries is 500 ppm, thus the maximum range for this industry would be 750 ppm and the calibrating gases would be 30% and 60% of 750 ppm--or approximately 225 ppm and 450 ppm. The range of the analyzer selected should give the lowest possible high-end reading without being lower than the span gas; for example, an analyzer with ranges of 0 to 500 ppm, 0 to 1000 ppm, and 0 to 2000 ppm would be calibrated on the 0 to 1000 ppm range for 750 ppm CO.

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Because some calibration gases with certificate of analysis have shown significant errors when compared with standard gases, good quality control procedures require the gas manufacturer to perform traceability analysis using NBS-Standard Reference Materials (SRM) or gas manufacturer's Certified Reference Materials (CRM). The EPA Traceability Protocol No. 1 should be required of the gas manufacturer for traceability analysis. This protocol is described in Section 3.0.4 of this Handbook. For convenience, a summary of Protocol No. 1 as it applies to standards of CO in N<sub>2</sub> is shown in Section 3.8.9. A list of gas manufacturer's that have prepared approved CRM's is available from EPA at the following address:

U.S. Environmental Protection Agency  
Quality Assurance Division (MD-77)  
Research Triangle Park, North Carolina 27711  
Attn: List of CRM Manufacturers

Calibration gases must be certified by the gas manufacturer to within  $\pm 2\%$  of the specified concentration. Do not store gas cylinders in areas subject to extreme temperature changes. Before each calibration, check the cylinder pressure of each calibration gas and replace any with  $< 1400 \text{ KN/m}^2$  (200 psi) pressure.

1.3.2 Silica Gel - Indicating-type 6 to 16 mesh should be dried at 175°C (347°F) for at least 2 h. The color should be blue when the water has been removed.

1.3.3 Ascarite - Ascarite (20 to 30 mesh) is commercially available. It consists of asbestos coated with sodium hydroxide which forms sodium carbonate when exposed to CO<sub>2</sub>. Eventually, the ascarite is spent, and has to be replaced since it cannot be regenerated.

TABLE 1.1. ACTIVITY MATRIX FOR PROCUREMENT OF APPARATUS AND SUPPLIES

Apparatus and supplies	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
<u>Sampling</u>			
Sampling probe	Stainless steel (316) or sheathed Pyrex glass with filter to remove particulate matter; no damage, cracks or breaks; leak free at 380 mm (15 in.) Hg	Visually check for length and composition ordered; leak check	Repair or return to supplier
Air-cooled condenser	Capacity sufficient to collect all condensed moisture from the sample gas; no damage, cracks, or breaks, leak free at 25 mm (1 in.) H <sub>2</sub> O positive pressure and at 380 mm (15 in.) Hg vacuum	Check for size and damage; leak check	Repair or return to supplier
Needle valve	Stainless steel; capable of regulating the flow rate over the range of 0 to 1 l/min (0 to 0.035 ft <sup>3</sup> /min); leak-free fittings	Install in sampling train; check for proper operation; leak check	Repair or return to supplier
Vacuum pump	Leak-free diaphragm at 380 mm (15 in.) Hg or equivalent; capable of maintaining a flow rate of 1 l/min (0 to 0.035 ft <sup>3</sup> /min) at 380 mm (15 in.) Hg for 30 S	Leak check; check for damage and capability of maintaining desired flow rate	Return to supplier
Rate meter	Rotameter or equivalent; no cracks, flaws, or erratic behavior; measure gas flow in the range of 0 - 1 l/min (0 to 0.035 ft <sup>3</sup> /min); agree within ±5% of standard rate meter	Check for cracks and flaws and calibrate against a wet test meter (Sec. 3.8.2)	Return to supplier

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TABLE 1.1 (continued)

Apparatus and supplies	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Flexible bag	Tedlar or equivalent; capacity of 60 to 90 l (2 to 3 ft <sup>3</sup> ); leak free at 51 mm (2 in.) H <sub>2</sub> O for 24 h	Check for capacity, damage, correct fittings; leak check	Repair or replace
Pitot tube	Type S (Method 2, Sec 3.1.1)	Calibrate according to Sec 3.1.2	Repair or return to supplier
Wet test meter	Capable of measuring total volume with accuracy of ±1% at a flow rate of 1 l/min (0.035 ft <sup>3</sup> /min)	Upon assembly, leak check all connections; calibrate by liquid displacement (Sec 3.8.2)	Reject if damaged, behaves erratically or cannot be adjusted properly
Barometer	Capable of measuring atmospheric pressure to ±2.5 mm (0.1 in.) Hg	Check against a mercury-in-glass barometer or equivalent (Sec 3.8.2)	Determine correction factor, or reject
Vacuum gauge	0 to 760 mm Hg range ±25 mm (1.0 in.) at 380 mm (15 in.) Hg	Check against a U-tube mercury manometer upon receipt	Adjust or return to supplier
<u>Sample Analysis</u>			
CO analyzer	NDIR spectrometer or equivalent; meets specifications in Sec 3.8.10 Appendices A and B	Have supplier (1) demonstrate that it meets or exceeds performance specs, and (2) provide a strip chart record of runs	Return to supplier for repair, adjustment, or replacement
Drying tube	Capacity of at least 200 g of silica gel; leak free at 380 mm (15 in.) Hg	Check upon receipt for proper size; leak check	Repair or return to supplier

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TABLE 1.1 (continued)

Apparatus and supplies	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Filter	Type and size recommended by NDIR manufacturer, or glass fiber filter	Check upon receipt for proper size and type	Return to supplier
CO <sub>2</sub> removal tube	Contains 500 g of ascarite; leak free at 380 mm Hg (15 in.); may combine silica gel and CO <sub>2</sub> removal tubes (Sec 3.8.1)	Check upon receipt for proper size, fittings, and leak check	Return to supplier
Ice water bath	Sufficient size to contain drying and CO <sub>2</sub> removal tubes	Check upon receipt for proper size; leak check	Return to supplier
Recorder (optional)	Strip chart recorder with operating voltage compatible with NDIR or equivalent data logger	Check for damage and proper operation over entire voltage range	Return to supplier
Carbon dioxide analyzers	Orsat analyzer (Method 3, Sec 3.2)	Check according to Meth. 3, Sec 3.2	Return to supplier
<u>Reagents</u>			
Calibration gases	Certified by manufacturer to $\pm 2\%$ of specified concentration; traceability to NBS-SRM or CRM (CO in N <sub>2</sub> )	Traceability analysis required of the gas manufacturer using EPA traceability Protocol No. 1	Return to supplier
Silica gel	Indicating-type 6 to 16 mesh; blue in color	Dry at 175°C (347°F) for at least 2 h prior to use	Discard or return to supplier
Ascarite	20 to 30 mesh	Check label for correct type	Return to supplier

## 2.0 CALIBRATION OF APPARATUS

Calibration of the apparatus is most important for maintaining data quality. The calibration procedures are designed for the equipment specified by Method 10 and described in the previous section. Table 2.1 summarizes the quality assurance activities for calibration. All calibrations should be recorded on standardized forms and retained in a calibration log book.

### 2.1 Metering System

2.1.1 Wet Test Meter - The wet test meter must be calibrated and must have the proper capacity. For Method 10, the wet test meter should have a capacity of  $\geq 2$  l/min (0.070 ft<sup>3</sup>/min). No upper limit is placed on the capacity; however, a wet test meter dial should make at least one complete revolution at the specified flow rate for each of the three independent calibrations.

Wet test meters are calibrated by manufacturers to an accuracy of  $\pm 0.5\%$ . Calibration must be checked initially upon receipt and yearly thereafter. The following liquid positive displacement technique can be used to verify and adjust, if necessary, the accuracy of the wet test meter (Figure 2.1) to  $\pm 1\%$ .

1. Level the wet test meter by adjusting the legs until the bubble in the level located on the top of the meter is centered.

2. Adjust the water volume in the meter so that the pointer in the water level gauge just touches the meniscus

3. Adjust the manometer to zero by moving the scale or by adding water to the manometer.

4. Set up the apparatus and calibration system (Figure 2.1).

5. Fill the rigid-walled 5-gal jug with distilled water to below the air inlet tube. Allow the system to equilibrate to room temperature (about 24 h) before use.

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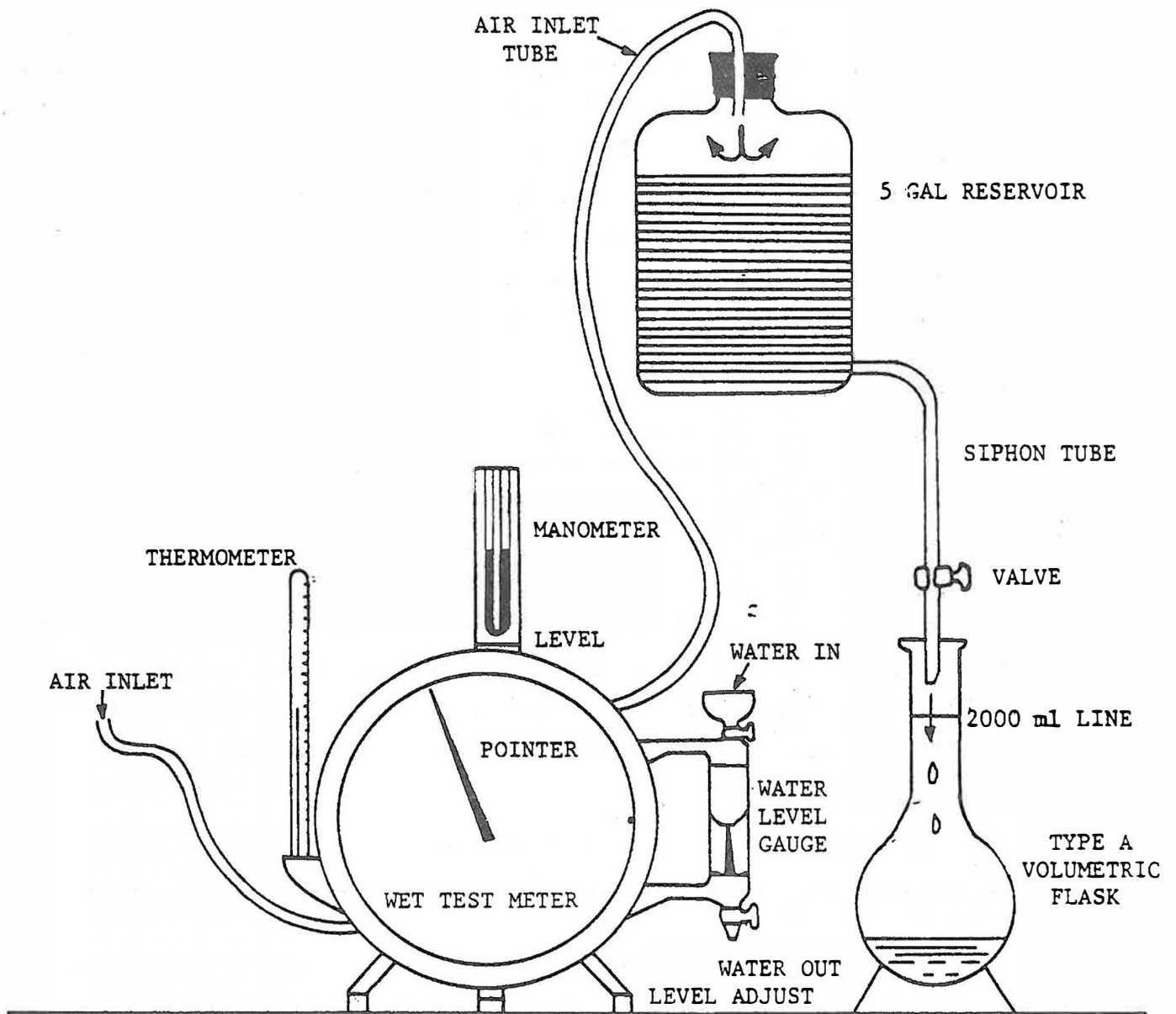


Figure 2.1. Calibration check apparatus for wet test meter.

6. Start water siphoning through the system, and collect the water in a 1-gal container, located in place of the class A volumetric flask.

7. Check the operation of the wet test meter as follows: If the manometer reading is  $<10$  mm (0.4 in.)  $H_2O$ , the meter is in proper working condition. Continue to step 8. If the reading is  $>10$  mm (0.4 in.)  $H_2O$ , the meter is defective; return it to the manufacturer for repair if the defect(s) (e.g., bad connections or joints) cannot be corrected.

8. Continue the operation until the 1-gal container is almost full and then plug the inlet to the saturator. If no leak exists, the flow of liquid to the container should stop; if the flow continues, correct for leaks. Turn the siphon system off by closing the valve, and then unplugging the inlet to the wet test meter.

9. Read the initial volume ( $V_i$ ) from the wet test meter dial, and record it on the calibration log, Figure 2.2.

10. Place a clean, dry volumetric flask (Class A) under the siphon tube, open the pinch clamp, and fill the flask to the mark. Note: The flask must be large enough to allow at least one complete revolution of the wet test meter dial with no more than two fillings of the flask.

11. Start the flow of water, be sure the flow of liquid is constant, and record the maximum wet test meter manometer reading during the test.

12. Carefully fill the volumetric flask, shut off the liquid flow at the 2-2 mark, and record the final volume ( $V_f$ ) from the wet test meter on Figure 2.2.

13. Perform steps 9 through 13 three times.

Since the water temperature in the wet test meter and the reservoir has been equilibrated to ambient temperature and since the pressure in the meter will equilibrate with that in the reservoir after the water flow is shut off, the air volume can be

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Wet test meter serial number 43-246 Date 1-27-80

Wet test meter flow range 0-120 lph Calibrated by JB

Volume of test flask,  $V_s$  2.00 l

Satisfactory leak check YES

Liquid in wet test meter and reservoir allowed to equilibrate with ambient temperature YES

Test number	Manometer reading, <sup>a</sup> mm H <sub>2</sub> O	Final volume ( $V_f$ ), l	Initial volume ( $V_i$ ), l	Total volume <sup>b</sup> ( $V_m$ ), l	Flask volume ( $V_s$ ), l	Percent error, <sup>c</sup> %
1	5	1.99	0	1.99	2.00	0.5
2	5	2.00	0	2.00	2.00	0
3	5	2.00	0	2.00	2.00	0

<sup>a</sup>Must be <10 mm (0.4 in.) H<sub>2</sub>O.

$${}^bV_m = V_f - V_i.$$

$${}^c\% \text{ error} = 100 (V_m - V_s) / V_s = \underline{0.5}, \text{ not to exceed } \pm 1\%.$$

Signature of calibration person Johnny Bench

Figure 2.2. Wet test meter calibration log.

compared directly to the liquid displacement volume. Any temperature or pressure difference would be less than measurement error and would not affect the final calculations.

The calibration error should not exceed  $\pm 1\%$ ; should this error of magnitude be exceeded, check all connections within the test apparatus for leaks, and gravimetrically check the volume of the standard flask. Repeat the calibration procedure, and if the tolerance level is not met, adjust the liquid level within the meter (see manufacturer's manual) until the specifications are met.

2.1.2 Rotameter - The Reference Method does not require calibration of the rotameter; however, besides cleaning and maintaining the rotameter according to manufacturer's instructions, its calibration curve and/or marking should be checked upon receipt and then after each test series. A procedure is as follows:

1. Prepare the apparatus (Figure 2.3) using short connections and tubing with the same ID used in the Method 10 sampling train.

2. Start the air flowing at  $0.5 \text{ l/min}$  ( $0.02 \text{ ft}^3/\text{min}$ ) to saturate the water in the wet test meter and to wet the interior surfaces of the wet test meter.

3. Record the barometric pressure ( $P_B$ ) on the rotameter calibration form (Figure 2.4A or 2.4B).

4. Adjust the flow ( $R_S$ ) to  $0.10 \text{ l/min}$  ( $0.0035 \text{ ft}^3/\text{min}$ ) with the needle valve.

5. Use a stopwatch to measure the time ( $\theta$ ) required to make at least two revolutions of the wet test meter dial.

6. Repeat step 4 with the flow ( $R_S$ ) adjusted to 0.25, 0.50, 0.75, and  $1.0 \text{ l/min}$  ( $0.009$ ,  $0.018$ ,  $0.027$ , and  $0.035 \text{ ft}^3/\text{min}$ ) going from a flow of  $1.0$  to  $0.10 \text{ l/min}$ . Record the time, the rotameter reading ( $R_S$ ), the elapsed time of the run ( $\theta$ ), the temperature of the liquid in the wet test meter ( $t_w$ ), the manometer reading at the wet test meter ( $D_m$ ), and the total volume displaced for each run ( $V_w$ ).

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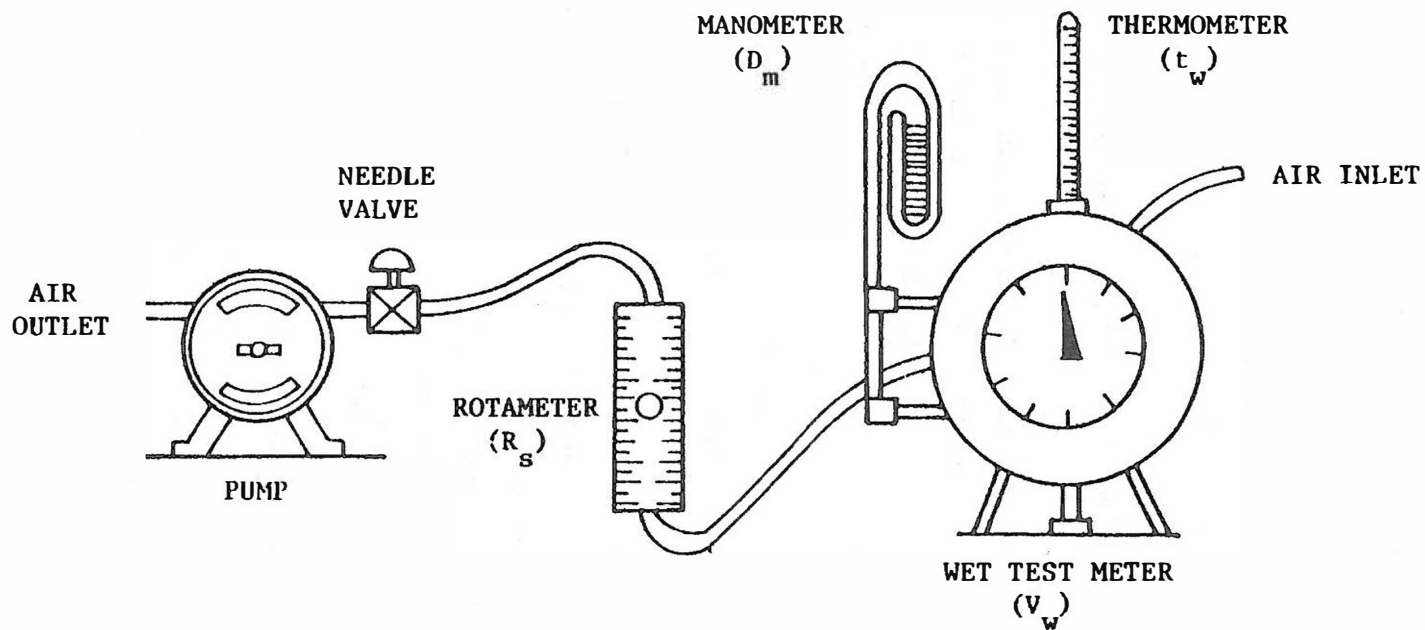


Figure 2.3. Rotameter calibration assembly.

Rotameter serial number 264572

Wet test meter number 43-246

Location SOURCE TEST LAB

Date 1-27-80

Barometric pressure,  $P_B$  29.9 in. Hg

Calibrated by JB

$R_s$ , ft <sup>3</sup> /min	$\theta$ , min	$t_w$ , °F	$D_m$ , in. H <sub>2</sub> O	$V_w$ , ft <sup>3</sup>	$V_r$ , ft <sup>3</sup>	$Q_s$ , ft <sup>3</sup> /min
0.027	3	68	-0.1	0.081	0.081	0.027

$R_s$  = rotameter setting, ft<sup>3</sup>/min (e. g., 0.009, 0.018, 0.027)

$\theta$  = time of calibration run, min

$t_w$  = temperature of the gas in wet test meter, °F

$D_m$  = pressure drop on the wet test meter, in. H<sub>2</sub>O (a negative number if calibrated as in Figure 2.3)

$V_w$  = gas volume passing through wet test meter, ft<sup>3</sup>

$V_r$  = gas volume passing through the rotameter corrected to STP, ft<sup>3</sup>

$Q_s$  = flow rate through rotameter, corrected to STP, ft<sup>3</sup>/min

$t_s$  = standard temperature, 68°F

$P_s$  = standard pressure, 29.92 in. Hg

$$V_r = \frac{V_w (P_B + D_m/13.6) (t_s + 460)}{(t_w + 460) P_s} = \frac{17.65 V_w (P_B + D_m/13.6)}{(t_w + 460)} = \text{ft}^3 \text{ at STP.}$$

$$Q_s = \frac{V_r}{\theta} = \text{ft}^3/\text{min at STP.}$$

Figure 2.4A. Rotameter calibration data form (English units).

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Rotameter serial number 264572

Wet test meter number 43-246

Location SOURCE TEST LAB

Date 1-27-80

Barometric pressure,  $P_B$  29.9 mm Hg

Calibrated by J.B.

$R_s'$ ℓ/min	$\theta$ , min	$t_w'$ , °C	$D_m'$ , mm H <sub>2</sub> O	$V_w'$ , ℓ	$V_r'$ , ℓ	$Q_s'$ , ℓ/min
0.75	5	20	-2.54	2.25	2.25	0.75

$R_s$  = rotameter setting, ℓ/min (e.g., 0, 0.50, 0.75)

$\theta$  = time of calibration run, min

$t_w$  = temperature of the gas in the test meter, °C

$D_m$  = pressure drop on the wet test meter, mm H<sub>2</sub>O (a negative number if calibrated as in Figure 2.3)

$V_w$  = gas volume passing through wet test meter, ℓ

$V_r$  = gas volume passing through the rotameter corrected to STP, ℓ

$Q_s$  = flow rate through rotameter, corrected to STP, ℓ/min

$t_s$  = standard temperature, 20°C

$P_s$  = standard pressure, 760 mm Hg

$$V_r = \frac{V_w (P_B + D_m/13.6) (t_s + 273)}{(t_w + 273) P_s} = \frac{0.386 V_w (P_B + D_m/13.6)}{(t_w + 273)} = \ell \text{ at STP.}$$

$$Q_s = \frac{V_r}{\theta} = \ell/\text{min at STP.}$$

Figure 2.4B. Rotameter calibration data form (Metric units).

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7. Calculate the volume ( $V_r$ ) at standard conditions (STP) for each test point, and record on Figure 2.4A or B.

8. Calculate the standard flow rate ( $Q_s$ ) for each test point, and record on Figure 2.4A or 2.4B.

9. Plot the rotameter setting ( $R_s$ ) versus the flow rate ( $Q_s$ ) on linear graph paper by using a flexible rule to construct a best-fit smooth curve through the data points. Note: A typical relationship is shown in Figure 2.5. All data points should be within  $\pm 2\%$  of the best-fit curve.

10. Apply the following corrections to convert the flow rate to STP if the rotameter is used in a field location where the barometric pressure and/or temperature is different from those recorded when the rotameter was calibrated.

$$Q_s = Q_f \left( \frac{P_f}{760} \times \frac{293}{t_f} \right)^{1/2} \quad \text{Equation 2-1}$$

where

$Q_s$  = flow rate corrected from field conditions to STP,  
l/min;

$Q_f$  = flow rate at field conditions from calibration curve,  
l/min;

$P_f$  = barometric pressure at field conditions, mm Hg; and

$t_f$  = temperature at field conditions, average temperature of  
sampling train, K.

2.1.3 Barometer - The field barometer should be adjusted upon receipt and before each test series to  $\pm 2.5$  mm (0.1 in.) Hg of a mercury-in-glass barometer. If a field barometer is not available, a nearby weather service barometric pressure can be used.

If the sampling point is higher in elevation than the weather station, the reported barometric pressure is reduced at a rate of 2.5 mm Hg/30 m (0.1 in. Hg/100 ft) of elevation difference; if the sampling point is lower than the weather station, the pressure should be increased at the same rate. Note: Make sure that the pressure obtained from the weather station has not been corrected to sea level conditions.

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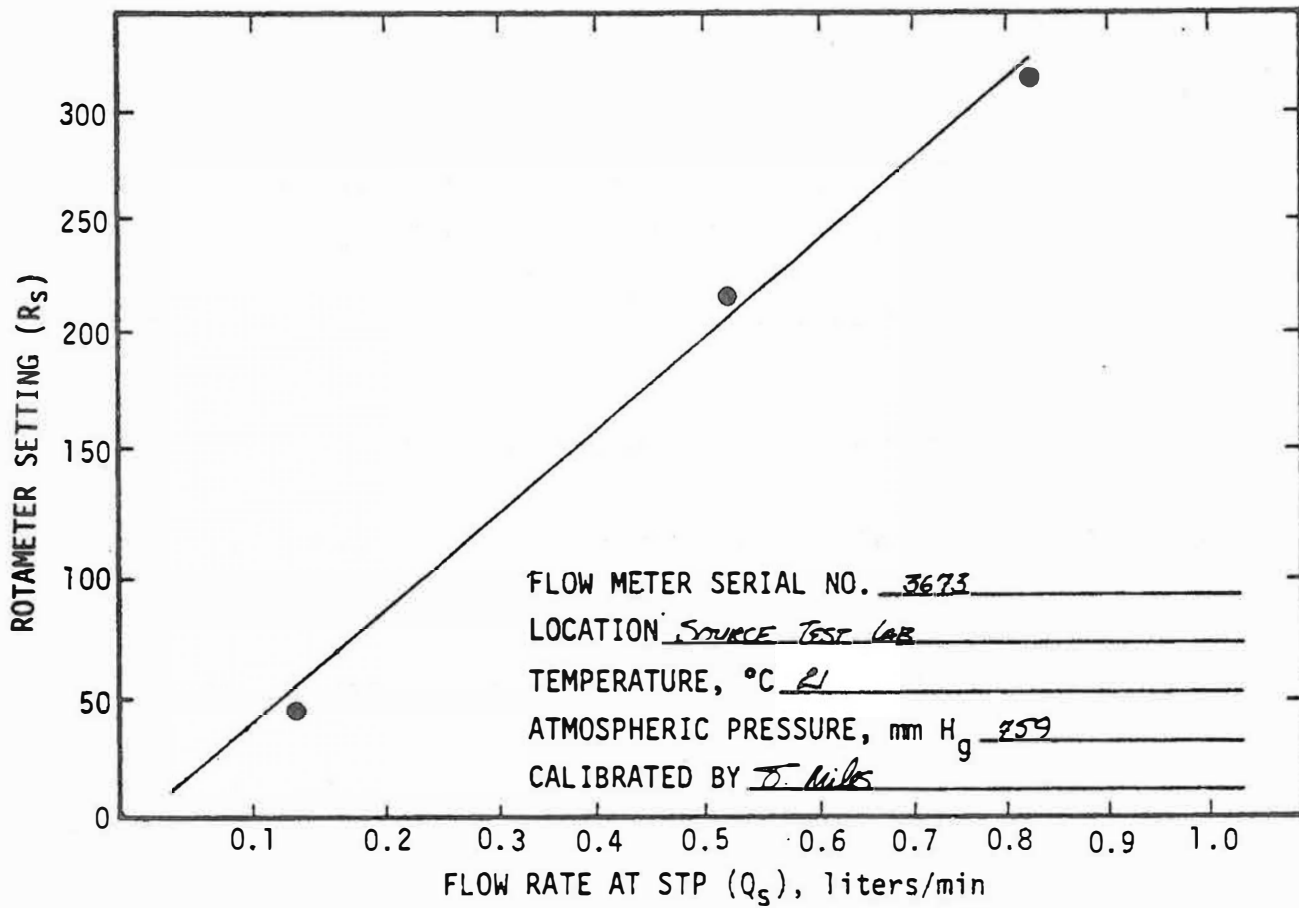


Figure 2.5. Typical rotameter calibration curve.

## 2.2 Analysis System

2.2.1 Carbon Monoxide Analyzer - The NDIR and the associated apparatus are shown in Figure 2.6; all components are the same as those used in the sampling train. Prior to leak check and calibration, add new or reconditioned indicating-type silica gel and new ascarite. Then leak check the system by closing the gas cylinder valves and excess flow valve; opening the control needle valve; and turning on the pump. If the rate meter at the NDIR inlet indicates flow, there is a leak. Check all connections and fittings for tightness. After the leak check, open the excess flow valve, shut off the pump, turn on the power, and allow the analyzer to warm up according to the manufacturer's instructions. Because the NDIR analyzer is temperature sensitive, allow it to warm up  $\geq 2$  h. When calibrating an analyzer, follow the manufacturer's instructions for setting the zero and the up-scale span point.

Calibration of NDIR analyzers may be multipoint checks; zero and span checks; or a zero check. Multipoint calibration is used to establish the calibration curve (or relationship) between the analyzer output and the CO input; this type of calibration is performed upon receipt of the analyzer, before any sampling series, and immediately after maintenance or internal adjustments of the analyzer. Zero and span checks establish whether the predetermined calibration curve has changed during analysis; this type calibration is performed at the end of each test series or at the start and end of each day for continuous sampling that runs for more than a day. A zero check is used both to establish whether the analyzer zero has drifted during a test and to adjust the analyzer if it has drifted. The zero check is made before each sample bag is analyzed for integrated samples.

Multipoint Calibration - The multipoint procedure can be used for introducing calibration gases to the analyzer and for plotting calibration curves.

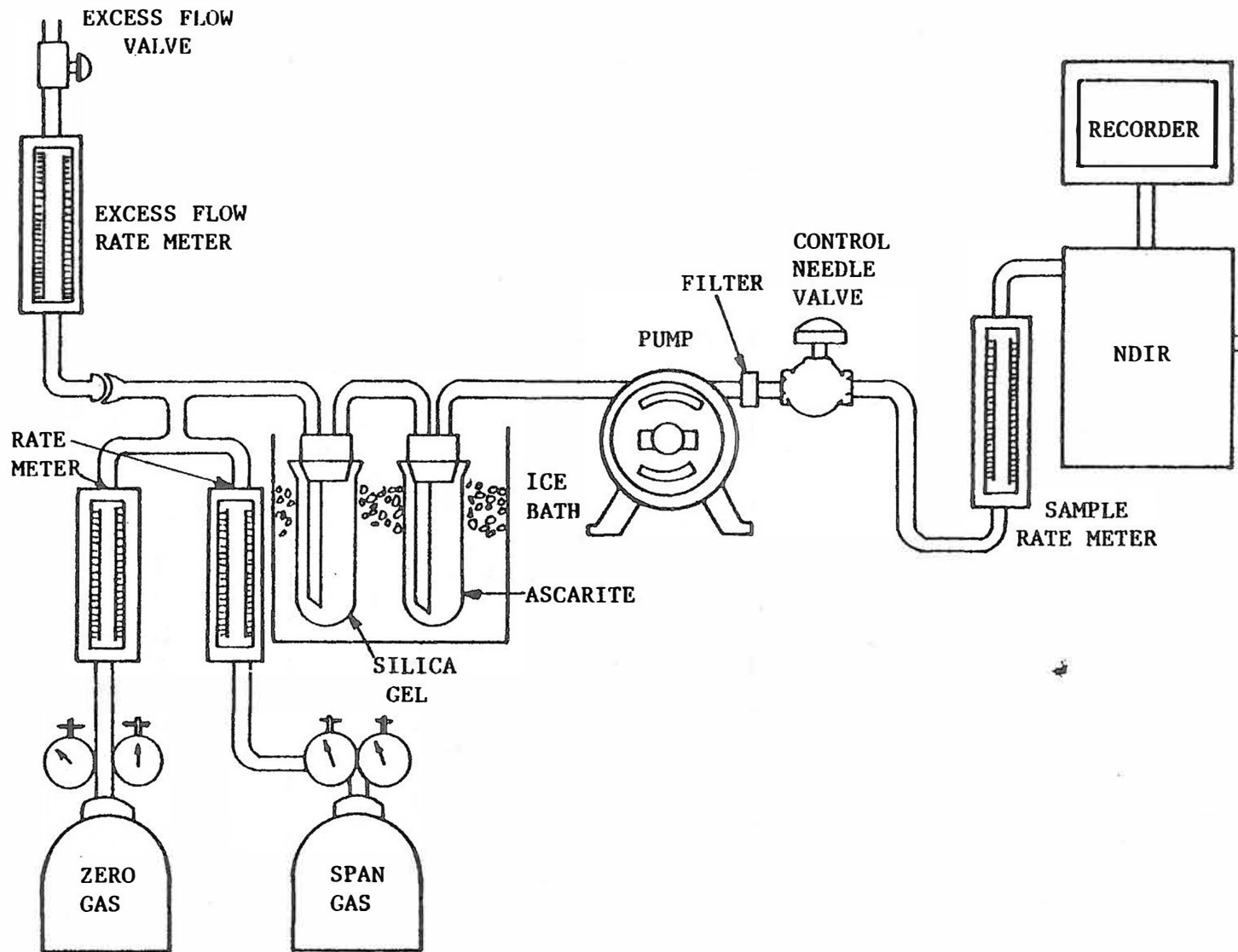


Figure 2.6. Calibration setup.

1. Open the excess flow valve and the control needle valve, and then turn on the pump.

2. Open the zero gas cylinder valve, and adjust the secondary pressure regulator to deliver 10 pounds per square inch, gauge (10 psig).

3. Adjust the zero gas control and the control needle valves slowly and simultaneously until the excess flow meter indicates a low rate (to ensure that air is not being pulled back through the excess flow line by the pump) and until the derived flow rate is reached at the sample rate meter. Note: Flow rates of 0.5 to 1.0  $\ell/\text{min}$  are normally recommended; most analyzers are not sensitive to flow rate changes below 1.0  $\ell/\text{min}$ , but the rate established at calibration should be maintained throughout the test series.

4. Set the analyzer zero by manufacturer's instructions after a stable reading is established (a minimum of 5 min).

5. Adjust the recorder zero control knob until the trace corresponds to the line representing 5% of the strip chart width above the chart zero or baseline to allow for any negative zero drift. If the strip chart already has an elevated baseline, use it for the zero setting.

6. Mark the strip chart trace at adjusted zero, and record the data on Figure 2.7.

7. Turn off the zero gas.

8. Open the span gas cylinder valve, and adjust the secondary pressure regulator to deliver 10 psig.

9. Open the span gas control valve until the excess flow meter indicates a low flow (refer to step 3).

10. Check the sample rate meter to assure that the same flow rate used to zero the analyzer is maintained; if not, adjust the flow valves.

11. Set the analyzer span by manufacturer's instructions after a stable trace is established (a minimum of 5 min). Note: Some analyzers require two or more adjustments of the zero and the span setting to get desired readings.

Location SOURCE TEST LAB Date 7-26-80 Operator T. WILSON  
Analyzer number 267-L Range 0-1000ppm Flow rate 1000 cc/min Cell pressure \_\_\_\_\_  
Zero gas N<sub>2</sub> Cylinder pressure 1500 PSIG Cylinder number 17674N  
Span gas 740 Cylinder pressure 1450 PSIG Cylinder number 15473  
60% span gas 445 ppm Cylinder pressure 1500 PSIG Cylinder number 15477  
30% span gas 225 ppm Cylinder pressure 1500 PSIG Cylinder number 15476  
Zero control setting 54 Span control setting 192  
Recorder type \_\_\_\_\_ Serial number 16759-G

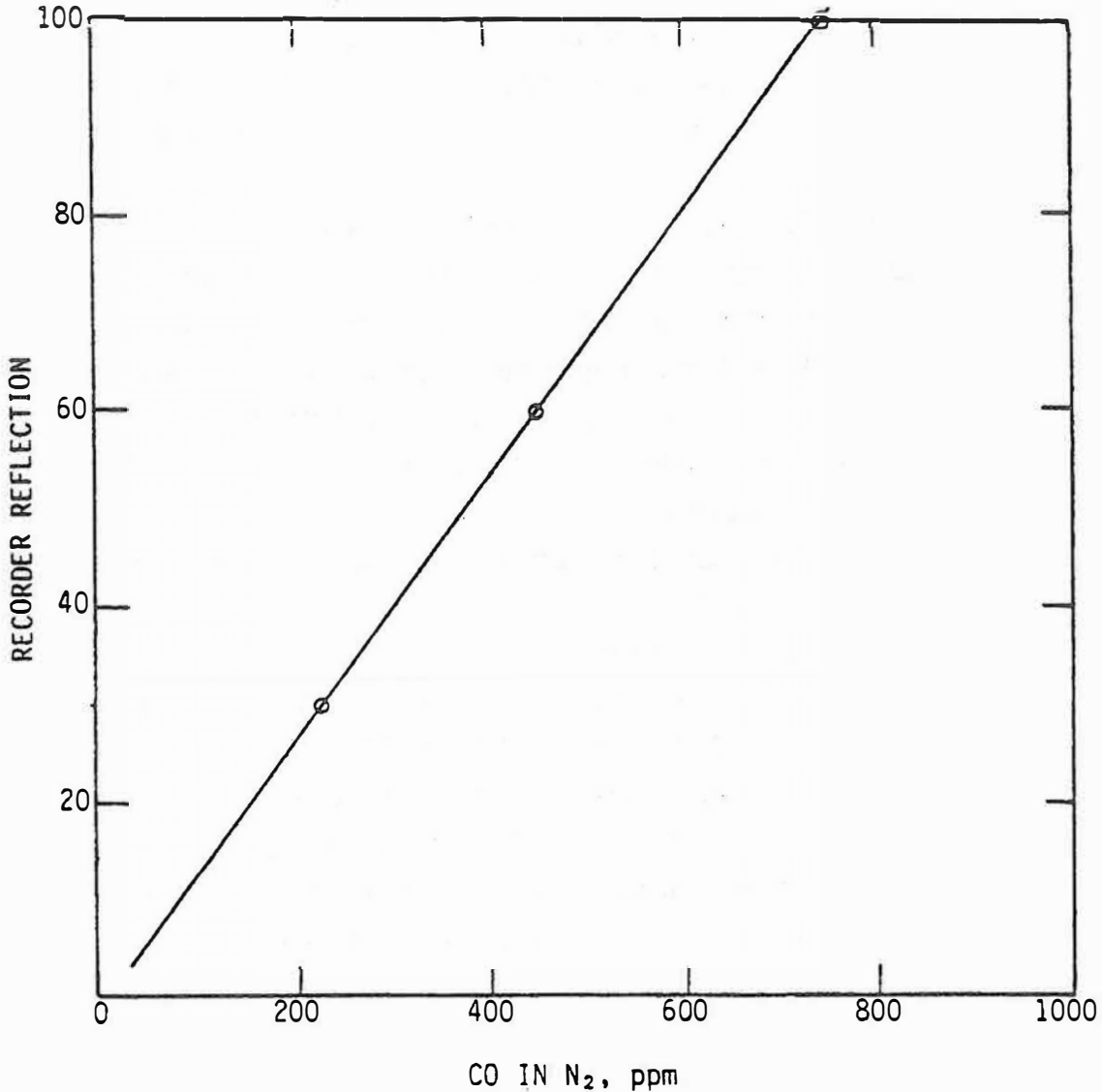


Figure 2.7. Sample calibration curve.

12. Mark the strip chart trace for the adjusted span, and record the data on Figure 2.7.

13. Close the span gas cylinder valve and the control valve, and remove the cylinder from the pressure regulator.

14. Replace the span gas cylinder with the reference gas cylinder that contains the 60% of span concentration.

15. Open the reference gas cylinder valve, and adjust the cylinder secondary pressure regulator to deliver 10 psig. Repeat steps 9 and 10.

16. Allow a stable trace to be established on the recorder. DO NOT ADJUST ANALYZER ZERO OR SPAN CONTROL. Mark the strip chart trace, and record the data on the form.

17. Repeat steps 13 through 16 for the 30% span concentration.

18. Plot concentration-versus-percentage relationship (Figure 2.7) after the multipoint calibration is complete.

19. Turn off all gas cylinders, and remove excess flow valve and rate meter assembly.

The analyzer is now ready for sample analysis.

Zero and Span Checks - Follow this procedure in conducting the zero and span checks:

1. Attach the excess flow rate meter and excess flow valve assembly as shown in Figure 2.6.

2. Open the zero gas cylinder valve, and adjust the secondary pressure regulator to deliver  $6.9 \times 10^4$  Pa gauge (10 psig).

3. Adjust the zero gas control and the control needle valves slowly and simultaneously until the excess flow meter indicates a low flow and the sample rate meter reads the same as it did during sampling.

4. Mark the strip chart trace as "unadjusted zero" and record the data on Figure 2.8 after a stable zero trace is established.

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Location Source Test LAB Operator T. Wilson  
 Analyzer 264-L Date 7-26-80

Time	Test number	Zero		Span		Percent difference
		unadjusted	adjusted	unadjusted	adjusted	
11:15	NR-1	-0.5%	-0-	99%	100%	1%

Figure 2.8. Example calibration verification record chart.

5. Adjust the analyzer zero to the zero reading established in the multipoint calibration; mark the strip chart trace as "adjusted zero"; and record the value on the form.

6. Turn off the zero gas.

7. Open the span gas cylinder valve, and adjust the secondary pressure regulator to deliver 10 psig.

8. Open the span gas control valve until the excess flow meter indicates a flow.

9. Check the sample rate meter and be sure that the same flow rate used to zero the analyzer is maintained. If not, adjust the flow valves.

10. After a stable span trace is achieved, label as "unadjusted span" and record the value on Figure 2.8.

11. If the unadjusted span value (from the calibration curve) differs from the span value determined during the multipoint calibration by more than  $\pm 10\%$ , reset the span setting. Use Equation 2-2 to determine the percentage difference between the two values.

$$\% \text{ difference} = \frac{CO_c - CO_s}{CO_s} \times 100 \quad \text{Equation 2-2}$$

where

$CO_c$  = concentration of span gas, and

$CO_s$  = concentration of span gas as determined.

Zero Check - A zero check is needed at the beginning of each integrated sample run and at the beginning and end of each continuous sampling test. Use the zero check and adjustment described above. Record the zero check values on Figure 2.8.

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TABLE 2.1. ACTIVITY MATRIX FOR CALIBRATION OF EQUIPMENT

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
<u>Metering System</u>			
Wet test meter	Capacity >2 l/min; accuracy $\pm 1.0\%$ for small wet test meter	Calibrate initially and quarterly by liquid displacement technique	Adjust until specifications are met, or return to manufacturer
Rotameter	Clean and maintain by manufacturer's instructions; calibrate to $\pm 2\%$	Upon receipt and after each field trip	Adjust and recalibrate, or reject
Barometer	$\pm 2.5$ mm (0.1 in.) Hg of mercury-in-glass barometer	Calibrate initially using a mercury-in-glass barometer and after each field test	Adjust to agree with certified barometer
<u>Analysis System</u>			
Pitot tube	Meth 2, Sec 3.1	Meth 2, Sec 3.1	Meth 2, Sec 3.1
CO analyzer	Multipoint calibration: 3-point (plus zero) to establish curve of analyzer	Calibrate upon receipt, at the beginning of any test series, and immediately after maintenance or internal adjustment, calibrate by adjusting span concentration to agree with certified gas concentration	Recalibrate instrument

### 3.0 PRESAMPLING OPERATIONS

The quality assurance activities for presampling operations are summarized in Table 3.1 at the end of this section. (See Section 3.0 of this Handbook for details on preliminary site visits.) The Pretest Sampling Checks, Figure 3.1, and Pretest Preparation Checklist, Figure 3.2, should be completed before leaving for the field test.

#### 3.1 Checking and Calibrating the Apparatus

3.1.1 Sampling Train - The CO sampling trains, integrated and continuous, are depicted in Figures 1.1 and 1.2, respectively. Commercial models are available, or sampling trains can be manufactured in house if the apparatus complies with specifications in the Reference Method (Section 3.8.10).

3.1.2 Probe - The probe should be cleaned internally by brushing first with tap water, then with deionized distilled water, and finally with acetone. Allow the probe to air dry. The objective is to leave the probe contaminant free.

The probe should be sealed and then leak checked at a vacuum of 380 mm (15 in.) Hg. See Section 3.8.1 for leak-check procedures.

3.1.3 Air-Cooled Condenser - The air-cooled condenser should be cleaned with tap water, then with deionized distilled water, and finally rinsed with acetone and allowed to air dry. The objective is to leave the condenser contaminant free. Leak check the condenser as described in Section 3.8.1.

3.1.4 Needle Valve and Rotameter - The metering valve and rotameter should be cleaned according to the manufacturer's recommendations prior to each field trip or on any sign of erratic behavior. After the rotameter is cleaned, it should be recalibrated (Section 3.8.2).

3.1.5 Vacuum Pump - The vacuum pump should be inspected for damage and leaks before each field trip. Leak test the pump as described in Section 3.8.1.

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Date 7-11-80 Completed by J. Williams

Pitot Tube

Identification number 763 Date 7-11-80  
Dimensional specifications checked?\*  yes  no  
Calibration required?  yes  no  
Date 7-11-80  $C_p$  0.84

Rotameter

Identification number 3674  
Calibration required?\*  yes  no

Barometer

Calibrated?\*  yes  no

\*Most significant items/parameters to be checked.

Figure 3.1. Pretest sampling checks.

Apparatus check	Acceptable		Quantity required	Ready		Loaded and packed
	Yes	No		Yes	No	
<u>Probe</u>						
Pyrex glass <input checked="" type="checkbox"/>	✓		4	✓		YES
Stainless steel						
Filter <u>GLASS WOOL</u>	✓		1 BOX	✓		YES
<u>Pitot tube</u>						
Type <u>5'</u>	✓		4	✓		YES
Length <u>5'</u>						
Calibrated* <u>YES</u>						
Differential pressure gauge <u>INCLINED MANOMETER</u>	✓		4	✓		YES
<u>Air-cooled condenser</u>						
Clean <u>YES</u>	✓		4	✓		YES
Leak checked* <u>YES</u>						
<u>Needle valve and rotameter</u>						
Clean <u>YES</u>	✓		4	✓		YES
Calibrated* <u>YES</u>						
<u>Barometer</u>						
Type <u>ANEROID</u>	✓		1	✓		YES
Calibrated* <u>YES</u>						
<u>Pump</u>						
Type <u>DIAPHRAGM</u>	✓		4	✓		YES
Leak checked <u>YES</u>	✓					
<u>Flexible bag</u>						
Type <u>TEDLAR</u>	✓		10	✓		YES
Leak checked* <u>YES</u>						
Evacuated* <u>YES</u>						

\*Most significant items/parameters to be checked.

Figure 3.2. Pretest preparation checklist.

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3.1.6 Flexible Bag - The flexible bag should be visually inspected for damage; leak checked; and evacuated before each field trip. For leak-check procedures, see Section 3.8.1.

3.1.7 Pitot Tube - The presampling operations required prior to using the pitot tube in the field are described in Method 2, Section 3.1.

3.1.8 Barometer - The field barometer should be calibrated prior to each field trip as described in Section 3.8.2.

### 3.2 Packing the Equipment for Shipment

3.2.1 Probe - Pack the probe in a case protected by polyethylene foam or other suitable packing material. Seal and protect the inlet and outlet of the probe from breakage. An ideal container is a wooden case (or equivalent) with separate polyethylene-lined compartments for individual pieces. The case should have handles or eyehooks that can withstand hoisting and that are rigid enough to prevent bending or twisting of the devices during shipping and handling.

3.2.2 Air-Cooled Condenser, Needle Valve, Rotameter, and Vacuum Pump - The air-cooled condenser, needle valve, rotameter, and vacuum pump should be mounted securely in a permanent container and cushioned (e.g., the pump bolted to the inside of a wooden box with rubber bushings between the pump and the box sides). Polyethylene foam can be used to cushion the components. The container should have handles or eyehooks that can withstand hoisting and that are rigid enough to prevent bending or twisting of components during shipping and handling.

3.2.3 Miscellaneous Equipment - Flexible bags, barometer, pitot tube, CO analyzer (if continuous sampling), and other miscellaneous equipment needed in the field should be packed conveniently and securely in containers and labeled (as to contents) for ease of identification in the field.

TABLE 3.1. ACTIVITY MATRIX FOR PRESAMPLING OPERATIONS

Operation	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
<u>Apparatus Check and Calibration</u>			
Probe	Free of contaminants; leak free	Clean internally by brushing, using tap water, deionized distilled water, and acetone, air dry; seal and check for leaks at 380 mm (15 in.) Hg prior to field use	Repeat cleaning procedure; repair or replace
Air-cooled condenser	Free of contaminants; leak free	As above	As above
Needle valve and rotameter	Clean and without signs of erratic behavior	Clean by manufacturer's recommendations prior to each field trip or at sign of erratic behavior	Repair or return to manufacturer
Vacuum pump	No damage or leaks; full oiler jar (if required)	Before field trip; visually inspect for damage; check oil level; leak check as described in Sec 3.8.1	Repair or replace
Flexible bag	Leak free and evacuated	Before field trip; leak check according to Sec 3.8.1, and evacuate	Repair or replace
Pitot tube	Meth 2, Sec 3.1	Meth 2, Sec 3.1	Meth 2, Sec 3.1
Barometer	Agrees $\pm 2.5$ mm (0.1 in.) Hg with mercury-in-glass barometer	Before field trip, calibrate against barometer (Sec 3.8.1)	Adjust and calibrate, or replace

(continued)

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TABLE 3.1 (continued)

Operation	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
<u>Packing Equipment for Shipment</u>  Probe	Rigid case; probe protected from breakage	Before field trip, pack probe in suitable container	Repack
Rotameter	Mounted in permanent protective container	Before field trip, mount permanently in protective container	Permanently mount in protective container
Flexible bag, pitot tube, CO analyzer, etc.	Packed in secure container	Before field trip, pack in shipping container	Repack

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#### 4.0 ON-SITE MEASUREMENTS

On-site measurement activities include transporting equipment to the test site; making duct measurements; conducting a velocity traverse; determining moisture content of stack gas; sampling for CO; recording data on appropriate forms; and labeling samples and containers for shipping. A clean "laboratory" type area free of excessive drafts should be designated for equipment storage, sample recovery, train assembly, and documentation.

Table 4.1 at the end of this section summarizes the quality assurance activities for on-site measurements. Copies of all field data forms mentioned in this section are in Section 3.8.12. The on-site measurements checklist, Figure 4.4 at the end of this section, provides the tester with a quick method for checking requirements during sampling.

##### 4.1 Equipment Transport

The most efficient means of transporting equipment from ground level to the sampling site should be decided during the preliminary site visit or through prior correspondence. Care should be taken to prevent injury to test personnel or damage to the test equipment during equipment transport.

##### 4.2 Sampling

The on-site sampling includes the following steps:

1. Preliminary measurements and setup.
  2. Preparation and setup of sampling train.
  3. Preparation of the probe (placing filter in probe).
  4. Connection to electric service.
  5. Leak check of the entire sampling train.
  6. Insertion of probe into the stack.
  7. Sealing of the port.
  8. Sampling (continuous or integrated).
  9. Determination of stack gas CO<sub>2</sub> content.
  10. Recording of the data.
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Upon completion of sampling, a leak check of the sampling train is required.

4.2.1 Preliminary Measurements and Setup - The sampling site should be selected in accordance with Method 1. If this is impossible due to duct configuration or other reasons, the sampling site location should be approved by the administrator. The site must be acceptable before a valid sample can be taken. Check for a 115-V, 20-A electrical service; this is adequate to operate the standard sampling train. Measure the stack ID. Either determine the minimum number of traverse points (Method 1) or check the points already determined during the preliminary site visit. Record all data on the point location form (Section 3.0). These measurements may be needed to locate the pitot tube and probe during sampling.

4.2.2 Stack Parameters - Determine the stack pressure and temperature; determine the range of velocity heads and the proper differential pressure gauge for the range; and conduct a leak check of the velocity pressure system (Method 2). Determine the approximate moisture content (Method 4 or its alternatives) for selecting the size of the condenser and the quantity of silica gel required in the sampling train. If the source has been tested before, an estimate of moisture based on previous test data should be sufficient.

Determine the dry molecular weight of the stack gas. If an integrated sample is required, follow the procedures for collecting the sample simultaneously with (and for the same length of time as) the CO sample, and use the sampling and analytical data forms in Method 3.

If a traverse is required, select a probe length sufficient for sampling all points. For large stacks, consider sampling from opposite sides of the stack to reduce the length of the probe. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack (or duct) for each sampling point.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the industry test procedures. Selection should assure that the sampling time per point (if traverse is required) is  $\geq 2$  min and that the sample volume corrected to standard conditions exceeds the minimum total gas sample volume required for that industry. The latter can be based on an approximate average sampling rate. The number of minutes sampled at each point (if a traverse is required) should be an integer or an integer plus one-half minute to avoid time-keeping errors. In some circumstances (e.g., batch cycles), it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas volumes; if so, the administrator's approval must first be obtained.

4.2.3 Probe and Sampling Train Preparations - Prepare the probe and the sampling train in the laboratory type area. First, place a loosely packed filter of glass wool in the end of the probe. (This filter should be changed after each test or after every 4 h of sampling.) Then if a continuous sample is required, fill the drying tube with silica gel (minimum of 200 g is recommended), and fill the CO<sub>2</sub> removal tube with ascarite. During preparation and assembly of the sampling train, keep all openings covered to prevent contamination. Just before collecting the sample, connect the probe and the flexible bag or NDIR analyzer to the sampling train.

4.2.4 Continuous Sampling - Follow the procedure below to obtain a continuous sample.

1. Leak check the train just before sampling by placing a gauge at the probe inlet and pulling a vacuum of  $\geq 250$  mm (10 in.) Hg. Turn the pump off. Note: The vacuum should remain stable for at least 30 s; if not, find and eliminate that leak before slowly releasing the vacuum gauge. This leak check is optional.

2. Connect the NDIR analyzer to electrical service and allow it to warm up according to the manufacturer's recommendations (minimum of 1 h). Whenever possible a 2-h warmup is preferred.

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3. Perform a multipoint calibration (Section 3.8.2).
4. Connect the NDIR to the sampling train as illustrated in Figure 4.1, and insert the probe into the stack at the predetermined sampling point.
5. Plug the sampling port to prevent dilution of the stack gas by in-leakage of ambient air.
6. Immediately adjust the gas flow rate to that recommended by the NDIR manufacturer [must be  $\leq 1$  l/min ( $0.035$  ft<sup>3</sup>/min)]. Purge the system by drawing at least 5 times the sampling system volume through the train or by drawing until the analyzer reading stabilizes.
7. Record the gas flow rate and the CO concentration on the field data form for continuous sampling (Figure 4.2).
8. Check the strip chart recorder (if used) for proper operation:
  - a. Chart speed control setting,
  - b. Gain control setting,
  - c. Ink trace readability,
  - d. Excess noise, and
  - e. Proper zero setting.
9. Determine the CO<sub>2</sub> concentration (Method 3) simultaneously with the CO monitoring.
10. Remove the probe from the stack, place a vacuum gauge at the probe inlet, perform the leak check (step 1), and record the leakage rate on the data form (Figure 4.2). This leak check is mandatory.
11. Disconnect the NDIR, and cap off both ends of the sampling train.
12. Perform the zero and span calibration (Section 3.8.2) upon completion of the testing, or once a day if continuous sampling lasts for more than one 24-h period.
13. Record the new zero and span settings in the comments section of the data form (Figure 4.2), and record the values on the strip chart recorder (if used).

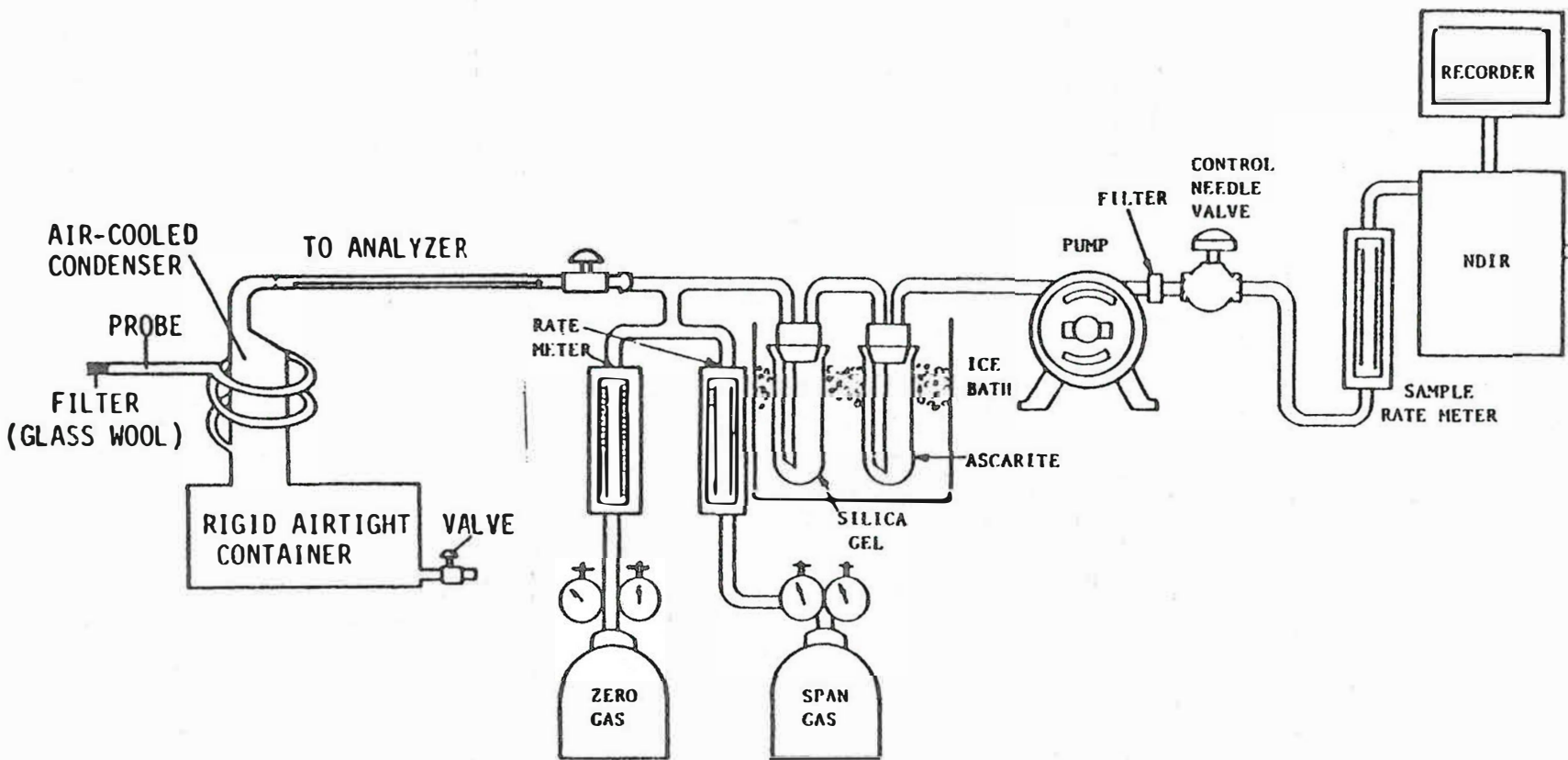


Figure 4.1. Continuous sampling apparatus.



4.2.5 Integrated Sampling - Integrated sampling is conducted at a rate proportional to the stack gas velocity, which has a linear relationship with the square root of the velocity head ( $\Delta P$ ). Following is a recommended method for determining proportional sampling rates:

1. Conduct a velocity traverse, and determine the maximum velocity head ( $\Delta P$  max) to be sampled.
2. Assign a sampling rate of 0.75  $\ell$ /min (0.03  $\text{ft}^3$ /min) to  $\Delta P$  max.
3. Determine the actual velocity head ( $\Delta P$ ).
4. Set the sampling flow rate using the following equation.

$$Q_s = Q_m \left( \frac{\Delta P}{\Delta P \text{ max}} \right)^{1/2} \quad \text{Equation 4-1}$$

where

$Q_m$  = maximum sampling rate, 0.75  $\ell$ /min (0.03  $\text{ft}^3$ /min),

$Q_s$  = sampling rate,  $\ell$ /min ( $\text{ft}^3$ /min),

$\Delta P$  = actual velocity head, mm (in.)  $\text{H}_2\text{O}$ , and

$\Delta P$  max = maximum velocity head, mm (in.)  $\text{H}_2\text{O}$ .

5. Determine the rotameter setting for the sampling rate ( $Q_s$ ) from the rotameter calibration curve, and adjust the rotameter accordingly.

Using this procedure will ensure that the sampling rate will not exceed 0.75  $\ell$ /min (0.03  $\text{ft}^3$ /min), and it will facilitate the preparation of a table or graph for easy reference prior to actual sampling.

Follow the procedure below to obtain an integrated sample.

1. Leak check the sampling train just prior to sampling by placing a U tube or inclined manometer at the probe inlet and pulling a vacuum of  $\geq 50$  mm (2 in.)  $\text{H}_2\text{O}$ . Turn the pump off.

Note: The vacuum should remain stable for at least 30 s. If a leak is found, repair before proceeding; if not, slowly release the vacuum gauge. This leak check is optional.

2. Disconnect the flexible bag from the sampling train, and insert the probe into the stack at the sampling point; if a

traverse is to be conducted, place the probe at the first point to be sampled.

3. Plug the sampling port to prevent dilution of the stack gas by in-leakage of ambient air.

4. Purge the system by turning on the pump and drawing at least 5 times the sampling system volume through the train, or purge for 10 min, whichever is greater.

5. Adjust the sample gas flow rate--not to exceed 1 l/min (0.035 ft<sup>3</sup>/min).

6. Connect the flexible bag to the sampling train (the connections should ensure a leak-free system), and begin sampling at a rate proportional to the stack gas velocity for the total sampling time specified by the standard of performance for the industry being sampled. The starting time for each test is when the sample bag is connected.

7. Record all data on the field sampling data form (Figure 4.3).

8. Determine the CO<sub>2</sub> concentration simultaneously with the CO monitoring. If enough volume will be collected in the flexible bag, an Orsat analysis for CO<sub>2</sub> concentration may be performed on the flexible bag used to collect the CO sample.

9. Disconnect and seal the flexible bag upon completion of sampling. Take care not to dilute the contents with ambient air.

10. Turn off the vacuum pump, remove the probe from the stack, and place a vacuum gauge at the probe inlet.

11. Repeat the leak check (step 1), and record the leakage rate on the data form (Figure 4.2). This leak check is mandatory.

12. Label each sample bag clearly and uniquely to identify it with its corresponding data form.

#### 4.3 Sample Recovery

Sample recovery should be performed in such a manner as to prevent contamination of the test sample and maintain sample integrity.

Plant name TRANS AMERICA INC Date 7-23-80  
 Sample location OUTLET STACK  
 Barometric pressure, ~~mm~~ (in.) Hg 29.20  
 Ambient temperature, °C (°F) 76° Stack temperature, °C (°F) 205  
 Initial leak check OK Final leak check OK  
 Operator T. WILSON

Sampling time, min	Clock time, 24 h	Traverse point	Velocity head ( $\Delta P_s$ ), <del>mm</del> (in.) H <sub>2</sub> O	Rotameter setting, l/min (ft <sup>3</sup> /min)	CO conc, ppm (dry basis)	CO <sub>2</sub> , %
5	10:00	1	0.25	0.56 l/min	45	9.5

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 Date January 4, 1982  
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Figure 4.3. Field sampling data form for CO (integrated sample).

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4.3.1 Continuous Sample Recovery - Continuous sampling for CO requires no sample recovery other than an integrated bag sample for CO<sub>2</sub>. The integrated method for determination of CO<sub>2</sub> content requires no sample recovery other than making sure the sample is labeled. The label should clearly and uniquely identify the sample with the test number, time of sampling and so forth. (Analysis of this sample is discussed in Method 3, Section 3.2.)

4.3.2 Integrated Sample Recovery - Integrated sample recovery for CO requires only that the gas bag be capped and properly labeled. If an integrated sample was obtained to determine the CO<sub>2</sub> content of the gas stream, it also should be capped and labeled. The labels should clearly and uniquely identify the test numbers, times of sampling, and so forth. The CO<sub>2</sub> sample could be the same as that to be analyzed for CO content. Analysis of the CO<sub>2</sub> sample is discussed in Method 3, Section 3.2.

#### 4.4 Sample Logistics (Data) and Equipment Packing

The sampling and the sample recovery procedures are followed until the required number of runs are completed. At completion, perform the following:

1. Return all samples to the base laboratory; check for proper labeling (time, date, location, number of each test, and other pertinent documentation).

2. Duplicate all data recorded during the field test by using carbon paper or by using data forms and a laboratory notebook. One set of data should be mailed to the base laboratory, given to another team member, or given to the agency; the other should be handcarried to prevent costly and embarrassing mistakes.

3. Examine all samples and sampling equipment for damage and properly pack for shipment to the base laboratory. Properly label all shipping containers to prevent loss of samples or equipment.

The postsampling operations--apparatus checks, sample analysis, and calculations--are discussed in the next two sections.

Continuous Sampling

Leak check prior to sampling (optional) yes  
NDIR allowed to warm up (1 h minimum)\* yes  
Multipoint calibration curve constructed\* yes  
Sample port plugged yes  
Sample flow rate properly set (manufacturer recommended and  
≤1 l/min)\* yes  
Sample system properly purged\* yes  
Posttest leak check (mandatory)\* yes  
All data properly recorded\* yes  
CO<sub>2</sub> concentration determined\* yes

Integrated Sampling

Sampling rate selected for integrated sampling \_\_\_\_\_  
Leak check prior to sampling (optional) \_\_\_\_\_  
Sample port plugged \_\_\_\_\_  
Sample train purged (5 times system volume or 10 min)\* \_\_\_\_\_  
Flexible bag properly sealed and labeled\* \_\_\_\_\_  
Posttest leak check (mandatory)\* \_\_\_\_\_  
All data properly recorded\* \_\_\_\_\_  
CO<sub>2</sub> concentration determined\* \_\_\_\_\_

\*Most significant item/parameters to be checked.

Figure 4.4. On-site measurements checklist.

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TABLE 4.1. ACTIVITY MATRIX FOR ON-SITE MEASUREMENTS

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Assembling sampling train	Meets specifications in Fig 1.1 or Fig 1.2	Before each sampling	Reassemble
Continuous sampling	Leak checked, no deflection on a vacuum gauge for a 30-s CO <sub>2</sub> determination, (Sec 3.8.1) leak check after sampling, (same as above)	Leak check the system; calibrate the NDIR before each test, and after each test run, or once a day (Sec 3.8.2); leak check after sampling (mandatory)	Correct the leak; repeat the sampling; re-calibrate; repeat the sampling
Integrated sampling	Leak checked, no deflection on a vacuum gauge for a 30-s period; CO <sub>2</sub> determination, sample volume, (Meth 3) minimum time and volume determined by applicable standard of performance; leak check after sampling (no deflection on a vacuum gauge for 30-s); sample proportionally to stack gas velocity for the specified length of time	Leak check the system; leak check after sampling (mandatory)	Correct the leak; repeat the sampling
Sample logistics (data) and packing of equipment	All data recorded correctly; all equipment checked for damage, and labeled for shipment; all sample containers properly labeled and packaged	After each sampling and before packing for shipment, visually check	Complete the data; repeat sample if damage occurred; correct if possible

## 5.0 POSTSAMPLING OPERATIONS

Table 5.1 at the end of this section summarizes the quality assurance activities for postsampling operations.

### 5.1 Apparatus Checks

Posttest checks must be made on most of the sampling apparatus. Record the data from the zero and span checks of the NDIR (continuous sampling only) and from the calibration, cleaning, and/or routine maintenance (Section 3.8.7) of the rotameter on Figure 5.1.

5.1.1 Rotameter - Calibration of the rotameter used during sampling should be verified by a posttest check which is similar to the initial calibration (Section 3.8.2) with the following variations:

1. The metering system should not have had any leaks corrected prior to the posttest check.

2. Only two flow rate checks need to be made. If the rotameter calibration factor ( $Y_r$ ) does not deviate  $>10\%$  from the initial calibration factor, the rotameter operation is acceptable; if it does, the rotameter should be cleaned and recalibrated (Section 3.8.2), but no calculations need be corrected. Record all required data on Figure 5.1.

5.1.2 NDIR Calibration Check (Continuous Sampling Only) - Calibration of the NDIR analyzer used during sampling must be checked upon completion of the testing period. Use the zero and span checks (Section 3.8.2). If the span check deviates more than  $\pm 10\%$  of the pretest span value, void all data back to the last acceptable calibration check.

### 5.2 Analysis Checks (Integrated Sampling Only)

The analyst should be familiar with the NDIR analyzer and its calibration procedure in order to obtain a precise and accurate analysis of samples and should use the analysis section of Figure 5.1 for a quick check of requirements during analysis of integrated samples.

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NDIR

Posttest zero check yes adjusted value 10%  
Posttest span check\* yes within  $\pm 10\%$  of pretest calibration  
Recalibration required? \_\_\_\_\_ yes  no  
If yes, void all data back to the last calibration check that  
was within the  $\pm 10\%$  limit

Rotameter

Pretest calibration factor,  $Y_r$  0.99 within  $\pm 5\%$   
Posttest check, \*  $Y_r$  0.99 within  $\pm 10\%$  of pretest  
Recalibration recommended? \_\_\_\_\_ yes  no  
If performed, recalibration factor,  $Y_r$  \_\_\_\_\_ ( $Y_r$  not used for  
emission calculations)  
Rotameter cleaned?  yes \_\_\_\_\_ no

Analysis (Integrated Samples)

Calibration gases traceable to NBS-SRM or CRM standard gas\* yes  
NDIR allowed to warm up (1 h minimum)\* yes  
Multipoint calibration curve constructed\* yes  
Sampling lines and analyzer properly purged (5 times system  
volume or 10 min)\* yes  
Three successive readings made from each bag yes  
Highest and lowest values differ by  $\leq 5\%$  yes

\*Most significant items/parameters to be checked.

Figure 5.1. Posttest sampling checks.

5.2.1 Calibration Gas Values - Concentrations of CO in the zero gas and in the calibration gases are determined as described in Section 3.8.2. The concentrations reported by the manufacturer should be traceable to an NBS-SRM or CRM standard gas.

5.2.2 NDIR Sample Values - After the analysis system is assembled according to Figure 2.6, use the following procedure to check the CO values:

1. Turn on the NDIR and allow it to warm up according to the manufacturer's recommendations (minimum of 1 h).

2. Perform a multipoint calibration (Section 3.8.2, Subsection 2.2.1).

3. Connect the flexible bag to the NDIR (Figure 5.2), and purge the sample lines and analyzer either by drawing at least 5 times the system volume through the system or by purging for 10 min whichever is greater.

4. Record at least three successive CO concentrations determined by the NDIR and the calibration curve (step 2). Note: The highest and lowest values should not differ by >5%.

5. Perform the zero and span checks (Section 3.8.2).

6. Repeat steps 3 through 5 for each bag sample.

Analyze the CO<sub>2</sub> integrated samples according to Method 3, Section 3.2.

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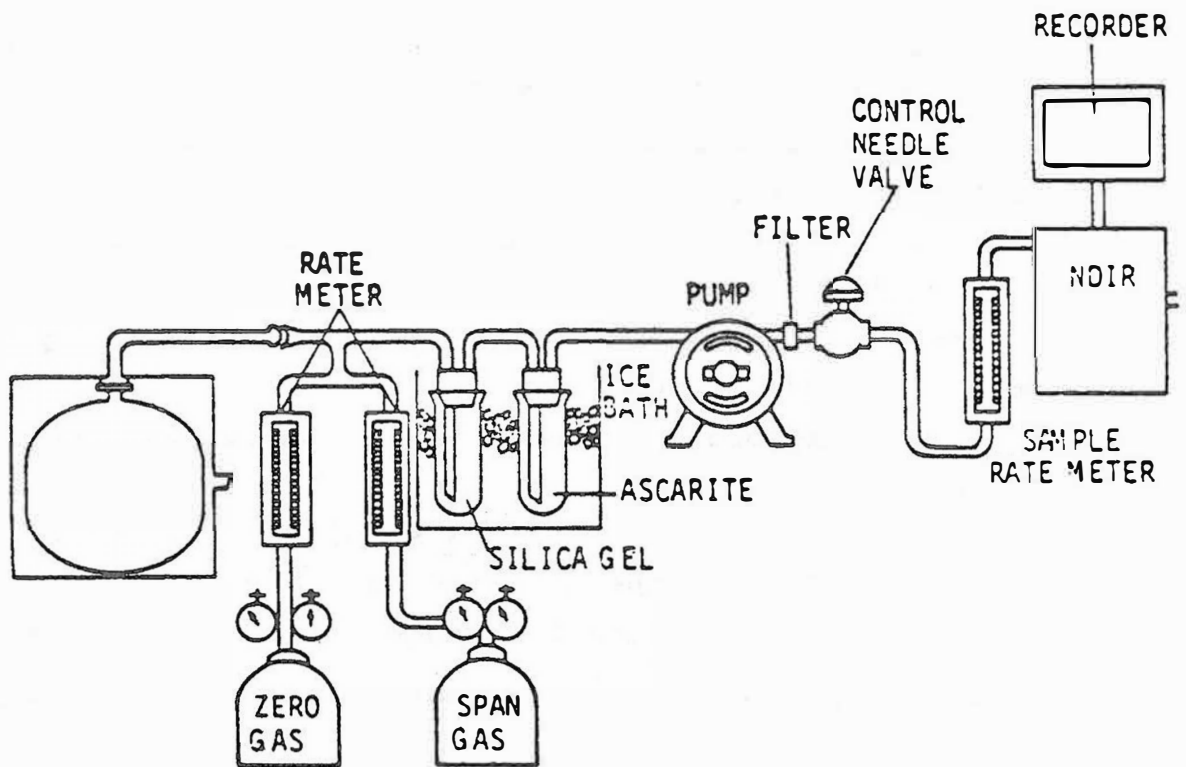


Figure 5.2. Integrated sample analytical apparatus.

TABLE 5.1. ACTIVITY MATRIX FOR POSTSAMPLING OPERATIONS

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
<u>Sampling</u>			
Rotameter	Within $\pm 10\%$ of desired flow rate	Make two independent checks (Sec 3.8.2)	Clean and recalibrate
NDIR (continuous sampling only)	Within $\pm 10\%$ of pretest calibration	After testing, check zero and span values (Sec 3.8.2)	Recalibrate and void all data back to last acceptable check
<u>Analysis (integrated sample only)</u>			
Calibration gases	Traceability to NBS-SRM or CRM performed by manufacturer according to Protocol No. 1	Sec 3.8.2	Return to supplier
Sample values	Values within 5% of each other	Make at least three successive determinations for each bag sample for CO and CO <sub>2</sub>	Repeat the analysis

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## 6.0 CALCULATIONS

Calculation errors due to mathematical mistakes can be a large part of total system error. Therefore, each set of calculations should be repeated or spotchecked by a team member other than the one who performed them originally. If a difference greater than a typical round-off error is detected, the calculations should be checked step by step until the source of error is found and corrected. Table 6.1 at the end of this section summarizes the quality assurance functions for calculations.

A computer program can be advantageous in reducing calculation errors. If a standardized computer program is used, check the original data entries; if differences are observed, make a new computer run.

Carryout calculations, retaining at least one decimal figure beyond that of the acquired data. Roundoff the final calculations to two significant digits for each run or sample in accordance with the ASTM 380-76 procedures. Record the results on Figure 6.1.

### 6.1 Nomenclature

The following nomenclature is used in the calculations.

$C_{CO_{stack}}$  = concentration of CO in stack, ppm by volume (dry basis),

$C_{CO_{NDIR}}$  = concentration of CO measured by NDIR analyzer, ppm by volume (dry basis),

$F_{CO_2}$  = volume fraction of  $CO_2$  in sample (i.e., % $CO_2$  from Orsat analysis divided by 100).

### 6.2 Calculations

The CO content (ppm) is measured by NDIR on a dry basis, after the  $CO_2$  content of the sample gas has been removed. This NDIR measurement must be corrected for the  $CO_2$  content (%) removed.

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Plant Trans America Inc. Date 7-23-80  
Sample location Outlet Stack  
Test number 1  
Bag number 101  
Operator J. Wilson

CO Concentration

$$C_{\text{CO}_{\text{NDIR}}} = \underline{40} \text{ ppm (dry basis).}$$

$$F_{\text{CO}_2} = \underline{9.0} \% \div 100 = \underline{0.09}.$$

$$C_{\text{CO}_{\text{stack}}} = C_{\text{CO}_{\text{NDIR}}} (1 - F_{\text{CO}_2}).$$

$$= \underline{40} (1 - \underline{0.090}) = \underline{36.4} \text{ ppm (dry basis).}$$

Errors:

Use Equation 6-1 and the data from Figure 6.1 to correct the NDIR reading.

$$C_{CO_{stack}} = C_{CO_{NDIR}} (1 - F_{CO_2}). \quad \text{Equation 6-1}$$

TABLE 6.1. ACTIVITY MATRIX FOR CALCULATION CHECKS

Characteristics	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Analysis data form	All data and calculations shown on Fig 6.1	Visually check	Complete the missing data
Calculations	Difference between check and original calculations less than or equal to round-off error	Repeat all calculations starting with raw data for hand calculations; check all raw data input for computer calculations, and hand calculate using one sample per test	Indicate any errors on Fig 6.1

(1111)

## 7.0 MAINTENANCE

Normal use of equipment subjects it to corrosive gases, temperature extremes, vibrations, and shocks. Keeping the equipment in good operating order over an extended period of time requires not only knowledge of the equipment but also routine maintenance. Maintenance of the entire sampling train should be performed quarterly or after 2830  $\ell$  (100  $\text{ft}^3$ ) of operation, whichever occurs first. Maintenance procedures for system components are summarized in Table 7.1. The following procedures are recommended, but not required, to increase the reliability of equipment.

### 7.1 Pumps

Several types of pumps are used in commercial sampling trains. Two of the most common are the fiber vane pump with in-line oiler and the diaphragm pump.

The fiber vane pump requires a periodic check of the oil and the oiler jar. The used oil (usually 10W nondetergent or machine weight) should be essentially the same translucent color as the unused or spare oil, and the jar should be no less than half full and leak free. When the fiber vane pump starts to run erratically or when the head is removed each year, the fiber vanes should be replaced.

The diaphragm pump requires little maintenance. If the pump runs erratically, it is normally due to a bad diaphragm or malfunctions in the valves; these parts are easily replaced and should be cleaned annually by complete disassembly of the train.

### 7.2 Rotameter

A rotameter should be disassembled and cleaned according to the manufacturer's instructions using the recommended cleaning fluids annually or more often if erratic behavior occurs.

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### 7.3 NDIR Analyzer

The CO analyzer comes with a manual that specifies maintenance procedures and how often each should be performed. Follow the manufacturer's recommendations, and call an experienced field service representative if internal adjustments are needed.

### 7.4 Other Sampling Train Components

All other sampling train components (probe, flexible bag, etc.) should be visually checked quarterly and completely disassembled and cleaned or replaced yearly. Many of the items (e.g., quick disconnects) should be replaced when damaged rather than checked periodically. Normally the best procedure in the field is to replace a component.

TABLE 7.1. ACTIVITY MATRIX FOR EQUIPMENT MAINTENANCE

Apparatus	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
Fiber vane pump	In-line oiler free of leaks and at least half full	Periodic check of oil and oiler jar; remove head and change fiber vanes yearly	Replace or refill oiler jar as needed
Diaphragm pump	Leak-free valves; diaphragm functioning properly	Clean valves during yearly disassembly or upon erratic behavior	Replace if cleaning will not correct the malfunction
Rotameter	Clean; no erratic behavior	Clean by manufacturer's recommendations yearly or upon erratic behavior	Replace
NDIR analyzer	Clean; no erratic behavior	Follow manufacturer's recommendations	Call service representative for expert repair
Sampling train components	No damage; no erratic behavior	Visually check every 3 mo; completely disassemble and clean or replace yearly	Repair or replace with spare component if in the field

## 8.0 AUDITING PROCEDURE

An "audit" is an independent assessment of data quality. Independence is achieved by using standards and equipment that are different from those used by a regular field crew. Although routine quality assurance checks conducted by a field team are necessary in generation of good quality data, they are not considered as part of the auditing procedure. Table 8.1 at the end of this section summarizes the quality assurance activities for auditing.

Based on the results of collaborative tests of Method 10,<sup>1</sup> two specific performance audits are recommended:

1. Audit of the analysis phase of Method 10.
2. Audit of data processing.

In addition to these performance audits, it is suggested that a system audit be conducted as specified by the quality assurance coordinator. The two performance audits and the system audit are described below in Subsections 8.1 and 8.2.

### 8.1 Performance Audits

Performance audits are conducted by the auditor to quantitatively assess the quality of data produced by the total measurement system (sample collection, sample analysis, and data processing). Due to the limited sizes of most emission-testing companies, it is recommended that these audits be performed by the responsible control agency once during every enforcement source test, regardless of whether the tests are conducted by agency or private company personnel. A source test for enforcement comprises a series of runs at one source.

8.1.1 Audit of Analysis Phase - An accuracy assessment should be made on the analytical phase by means of a cylinder gas audit as follows:

1. Challenge the analyzer with an audit gas of known concentration at two points. Audit Point 1 should be within 45 to 55% of analyzer full span and audit point 2 should be within 85 to 95% of analyzer full span.

Use a separate audit gas cylinder for audit points 1 and 2. Do not dilute gas from the audit cylinder when challenging the analyzer.

The analyzer should be challenged at each audit point for a sufficient period of time to assure adsorption-desorption of the system surfaces has stabilized.

2. Operate each analyzer in its normal sampling mode, i.e., pass the audit gas through all filter, scrubbers, conditioners, and other analyzer components used during normal sampling and as much of the sampling probe as is practical.

3. Use audit gases that have been certified by comparison to NBS-SRM or gas manufacturer's (CRM) following EPA Traceability Protocol No. 1. As an alternative to Protocol No. 1 audit gases, CRM may be used directly as audit gases. A list of gas manufacturers that have prepared approved CRM's is available from EPA at the following address:

U.S. Environmental Protection Agency  
Quality Assurance Division (MD-77)  
Research Triangle Park, North Carolina 27711  
Attn: List of CRM Manufacturers

The differences between the actual concentration of the audit gas and the concentration indicated by the analyzer is used to assess the percent accuracy (%A) of the test data as shown in Figure 8.1. If the %A for an audit point is not within limits, challenge the analyzer with the same concentration to verify the response.

The calculated %A should be within  $\pm 15\%$ . Results of the calculated %A should be included in the enforcement source test report as an assessment of accuracy of the analytical phase of Method 10 during the actual enforcement source test.

Tester R Hawks Source Richards Inc.  
 Analyzer Acme Model 461 Range 0-1000 ppm  
 Analyzer serial number 49-A75-C101  
 Auditor G Saunders Date 4/22/80  
 Cylinder ID EE 1591 Concentration 320, 582, 905  
 Remarks \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Audit cylinder conc $(C_{CO})_a$ , ppm	Analyzer response $(C_{CO})_m$ , ppm	Percent accuracy, (%A), %*
320	304	5.0
582	560	3.8
905	863	4.6

$$* \%A = \frac{(C_{CO})_m - (C_{CO})_a}{(C_{CO})_a} \times 100.$$

Figure 8.1. Example of an audit summary report.

(1117)

If the audit indicates that the analyzer is out of tolerance, corrective action must be taken.

If the analysis is to be performed in the laboratory (integrated sampling only), the above audit procedure can be used for a pretest audit (optional).

8.1.2 Audit of Data Processing - The data reduction process involves reading a strip chart record, calculating an average, and transcribing or recording it. The data thus obtained can be compared to the calibration curve as an independent check of the entire data reduction process or, the audit may be accomplished by providing the laboratory team with specific data (exactly as would occur in the field) and requesting that copies of the data reduction be returned to the evaluator.

When a difference between the originals and the audits on data reduction and calculations exceeds round-off error, all data from the source test should be checked, and the errors should be clearly explained to the team to prevent or minimize reoccurrences.

## 8.2 System Audit

A system audit is an on-site qualitative inspection and review of the quality assurance activities used by the test team to evaluate the total measurement system (sample collection, sample analysis, data processing, etc.). Initially, a system audit specified by a quality assurance coordinator should be conducted for each enforcement source test, which by definition comprises three runs at one source. After the team gains experience, the frequency of audit may be reduced--for example, to once every four tests.

The auditor (i.e., the person performing the system audit) should have extensive background experience in source sampling and more specifically, with the measurement system that he is auditing. The functions of the auditor are summarized in the following:

1. Observe procedures and techniques of the field team during sample collection.

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2. Check/verify the records of apparatus calibration and the quality control charts used in the laboratory analysis.

3. Record the results of the audit and forward them with comments on source team management to the quality assurance coordinator so that any needed corrective actions may be implemented.

The auditor should observe the field team's overall performance of the source test. Specific operations to observe should include, but not be limited to:

1. Setting up and leak testing the sampling train.
2. Use of proper zero and span gases.
3. Purging of sampling train.
4. Sampling rate (constant or proportional).
5. Sample recovery and preparations for shipment, if applicable.

Figure 8.2 is a suggested checklist form for use by the auditor in developing his/her own list of important techniques/steps to observe.

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			OPERATION
yes	no	comment	
<input checked="" type="checkbox"/> <input checked="" type="checkbox"/> <input checked="" type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<hr/> <hr/> <hr/>	<p style="text-align: center;"><u>Presampling Preparations</u></p> <ol style="list-style-type: none"> <li>1. Knowledge of process conditions</li> <li>2. Traceability of calibration gas established</li> <li>3. Calibration of pertinent equipment, in particular, the NDIR</li> </ol>
<input checked="" type="checkbox"/> <input checked="" type="checkbox"/> <input checked="" type="checkbox"/> <input checked="" type="checkbox"/> <input checked="" type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<hr/> <hr/> <hr/> <hr/> <hr/> <i>OK</i>	<p style="text-align: center;"><u>On-Site Measurements</u></p> <ol style="list-style-type: none"> <li>4. Leak test of sampling train</li> <li>5. NDIR warmup per manufacturer's recommendations</li> <li>6. Purging the train prior to sampling</li> <li>7. Proportional sampling</li> <li>8. Frequency of zero and span checks</li> <li>9. Drying agents checked and replaced frequently</li> </ol>
<input checked="" type="checkbox"/> <input checked="" type="checkbox"/> <input checked="" type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<hr/> <hr/> <hr/>	<p style="text-align: center;"><u>Postsampling</u></p> <ol style="list-style-type: none"> <li>10. Transfer and handling of sample</li> <li>11. Data reduction procedure/check</li> <li>12. Calibration checks</li> </ol>
GENERAL COMMENTS:			

Figure 8.2. Method 10 checklist to be used by auditors.

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TABLE 8.1. ACTIVITY MATRIX FOR AUDITING PROCEDURES

Audit	Acceptance limits	Frequency and method of measurement	Action if requirements are not met
<u>Performance Audit</u>  Analysis phase	$0.85 < \frac{(C_{CO})_m}{(C_{CO})_a} < 1.15$ $(C_{CO})_m = \text{analyzer response, ppm,}$ $(C_{CO})_a = \text{concentration of audit cylinder, ppm}$	Once during every source test, measure reference sample and compare to the true value	Review operating and calibration techniques/procedures
Data processing	Original and check results agree within roundoff error	Once during every source test; perform independent data reduction and calculations	Check and correct all data for the test series
<u>System Audit</u>  Observance of techniques	Operation technique as described in this section of the Handbook	Once during every source test until experience gained, and then, every fourth test, observe techniques; use audit checklist (Fig 8.2)	Explain to team any deviations from recommended techniques; record data and comments on Fig 8.2

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## 9.0 RECOMMENDED STANDARDS FOR ESTABLISHING TRACEABILITY

To achieve data of desired quality, two considerations are necessary:

1. The measurement process must be in a state of statistical control.
2. The systematic errors, when combined with the random variations (errors of measurement), must result in a suitably small uncertainty.

To ensure good data, it is necessary to perform quality control checks and independent audits of the measurement process; to document the data by quality control charts (as appropriate); and to use materials, instruments, and procedures which can be traced to a standard of reference.

The working calibration standards should be traceable to primary or higher level standards such as the two listed below.

1. A wet test meter (2-l/min capacity) with a  $\pm 1\%$  accuracy verified by liquid displacement, as described in Section 3.8.2.
2. NBS-SRM and gas manufacturers CRM gases are considered primary standards.

### 9.1 Traceability Protocol for Establishing True Concentration of CO Gases used for Calibration and Audit

The traceability protocol described in this section is intended to minimize systematic and random errors during the analysis of calibration and audit gas standards and to establish the true concentrations by means of National Bureau of Standards, Standard Reference Materials (NBS-SRM's) or certified reference materials or other NBS traceable gases.

Performance standards promulgated<sup>3,4,5</sup> by the U.S. Environmental Protection Agency (USEPA) for stationary sources require continuous monitoring systems for specified pollutants. Extractive continuous monitoring systems for gaseous pollutants must be calibrated and audited using gas standards that are accurate and

stable. Traceability requires direct comparisons between the calibration and audit gas standards and either an NBS-SRM or a certified reference material or a gas manufacturer's primary standard (GMPS) which is referenced to an NBS-SRM. All comparisons are made using an instrument calibrated with applicable NBS-SRM's. Traceability must be performed by the gas standard manufacturer at the time of purchase; reanalysis to verify traceability may be performed by the gas standard manufacturer or by the user.

9.2 Establishing Traceability of Commercial Cylinder Gases to NBS-SRM Cylinder Gases

The following procedures for periodic multipoint calibrations and daily instrument span checks are prescribed to minimize systematic errors. Separate procedures for instrument span checks are described for linear and nonlinear instruments. To be linear, the difference between the concentrations indicated by the calibration curve and the straight line drawn from the point determined by the zero gas to the highest point determined by calibration must not exceed 2% of full scale at any point on the curve. A list of NBS-SRM CO cylinder gases recommended for traceability of commercial cylinder gases is shown in Table 9.1.

TABLE 9.1. NBS-SRM CARBON MONOXIDE (CO) GASES AVAILABLE FOR TRACEABILITY AND AUDIT OF CO GAS STANDARDS

NBS-SRM number	Type	Size l at STP	Nominal concentrations
1677b	CO in N <sub>2</sub>	870	10 ppm
1678b	CO in N <sub>2</sub>	870	50 ppm
1679b	CO in N <sub>2</sub>	870	100 ppm
1780a	CO in N <sub>2</sub>	870	500 ppm
1681a	CO in N <sub>2</sub>	870	1000 ppm

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9.2.1 Multipoint Calibration - A multipoint calibration curve should be prepared monthly by using two SRM cylinder gases and the zero gas. The zero gas must contain  $\leq 0.2\%$  of the full-scale concentration of the component being analyzed, and it must be free of any impurity that will cause a response on the analytical instrument.

Multipoint calibration is accomplished by using a calibration flow system to dilute the SRM of highest concentration with the zero gas.

1. Read the responses for six points displaced from 0 to 100% of the instrument's full scale.
2. Plot the data, and draw the calibration curve.
3. Read the response for the SRM of lower concentration without dilution.
4. Compare the apparent concentrations from the calibration curve to the true concentration of the lower SRM. Note: If the difference between the apparent and the true is  $>3\%$  of the true concentration, repeat the multipoint calibration procedure.
5. Test the calibration curve for linearity. Proceed to either Subsection 9.2.2 or 9.2.3.

9.2.2 Instrument Span Check for Linear Responses - The span check should be performed at the start of each day that cylinder gases are to be analyzed.

1. Read the instrument's response to the highest SRM (or GMPS) in the range to be used and check the response to the zero gas.
2. Adjust the response to the value obtained in the most recent multipoint calibration, and proceed to Subsection 9.3. Note: Cylinder gases analyzed with a linear instrument must not have a concentration  $>15\%$  above the highest available SRM concentration.

9.2.3 Instrument Span Check for Nonlinear-Response - The span check should be performed at the start of each day that cylinder gases are to be analyzed.

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1. Read the instrument's responses to two SRM's (or GMPS's) in the range of calibration gases to be analyzed, and check responses to zero gas.

2. Set the instrument's zero with the zero gas, and adjust its response to the highest SRM (or GMPS) to the value obtained in the most recent multipoint calibration.

3. Read the response to the lower SRM (or GMPS). Note: If the response to the lower SRM (or GMPS) varies by >3% from the response in the most recent multipoint calibration, a full multipoint calibration must be performed (Subsection 9.2.1); otherwise, proceed to Subsection 9.3. Calibration gases analyzed with a nonlinear instrument must not have a concentration greater than the highest available SRM concentration.

### 9.3 Determining True Concentrations of Cylinder Gases

Direct comparison of the cylinder gas to an SRM (or GMPS) should compensate for variations in instrument responses between the daily span check and the analyses; significant variations in responses often result from changes in room temperature, line voltage, and so forth. Analyses in this procedure should be performed in triplicate (3 pairs) to expose erroneous data points and excessive random variations in instrument responses.

After the gas cylinder has been filled, wait a minimum of 4 days before beginning the following procedure. If necessary, adjust the instrument span prior to the analyses, but do not adjust it during the triplicate analyses.

1. Compare each cylinder gas directly with the nearest SRM (or GMPS) by taking alternate readings of the SRM and calibration gas responses in triplicate. Note: The response to zero gas must be read frequently so that the change in successive zero responses are not >1% of full scale.

2. For each of the six readings, determine the apparent concentration of the SRM (or GMPS) or cylinder gas by referring to the calibration curve.

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3. For each pair of readings--one SRM (or GMPS) and one cylinder gas, calculate the true concentration of the cylinder gas by using Equation 9-1:

True conc of cyl gas =

$$[\text{appar conc of cyl gas}] \times \left[ \frac{\text{true conc of SRM (or GMPS)}}{\text{appar conc of SRM (or GMPS)}} \right].$$

Equation 9-1

4. Determine the mean of the three values to get the true concentration of the cylinder gas. Note: If any one of the three values differs from the mean by >1.5%, discard the data, reset the instrument span (if necessary), and repeat steps 1 through 4.

#### 9.4 Using Gas Manufacturer's Primary Standards

Gas manufacturer's primary standards (GMPS) are gas mixtures prepared in pressurized containers and analyzed against SRM cylinder gases. Using GMPS's instead of SRM's will help to conserve SRM's where large quantities of cylinder gases are analyzed. A GMPS may be used for instrument span checks (Subsections 9.2.2 and 9.2.3) and for cylinder gas analyses (Subsection 9.3) if the following conditions are met.

1. A GMPS must have been analyzed against SRM cylinder gases as described in Subsections 9.2 and 9.3 within 30 days of their use for cylinder gas analysis, and should be compared on the days that instrument multipoint calibrations are performed.

2. A GMPS must not have changed in concentration by >1% per mo (average) for the 3-mo period prior to use for cylinder gas analysis.

In no case may a GMPS be substituted for an SRM in multipoint calibrations (Subsection 9.2.1).

#### 9.5 Verifying Cylinder Gas Stability

The stability of each cylinder gas should be verified by a second set of triplicate analyses (using the procedure in Subsection 9.3) a minimum of 7 days after the first set of triplicate

analyses. The mean of the second triplicate analyses must not differ from the mean of the first triplicate analysis by >1.5%.

#### 9.6 Reanalyzing Cylinder Gases

Either the gas manufacturer or the user must reanalyze the cylinder gas every 6 mo from the last analysis date by the procedure in Subsection 9.3. Cylinder gases used for audits may need to be analyzed more often than every 6 mo.

#### 9.7 Minimum Cylinder Pressure

No cylinder gas with pressure <200 psi, as shown by the cylinder gas regulator gauge, should be used.

#### 9.8 Labeling the Cylinder and Preparing the Analysis Report

Each gas cylinder should have the following minimum traceability information either on a gummed label affixed to the cylinder wall and/or on a tag attached to the cylinder valve:

1. Cylinder number.
2. Mean concentration of cylinder gas, ppm or mol%.
3. Balance gas used.
4. Last analysis date.
5. Expiration date (6 mo after last analysis date for reactive gases, and 12 mo after for diluent gases).

A written analysis report certifying that the cylinder gas has been analyzed according to the protocol described in this section should contain the following information:

1. Cylinder number.
2. Mean concentration of cylinder gas (ppm or mol%) on last analysis date.
3. Replicate analysis data.
4. Balance gas used.
5. Numbers of NBS-SRM's used.
6. Analytical principle used.
7. Last analysis date.

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The user should maintain a file of all analysis reports for 3 yr.

9.9 Conducting Performance Audits

The USEPA will initiate a national performance audit program of cylinder gases prepared by this protocol. Cylinder gases prepared following the protocol will be obtained (directly or indirectly) by the USEPA and analyzed in their laboratory for accuracy compared to the gas manufacturer's reported concentration.

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## 10.0 REFERENCE METHOD<sup>a</sup>

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

#### 1. Principle and Applicability.

1.1 *Principle.* An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 *Applicability.* This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

#### 2. Range and sensitivity.

2.1 *Range.* 0 to 1,000 ppm.

2.2 *Sensitivity.* Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences.* Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water (H<sub>2</sub>O) and carbon dioxide (CO<sub>2</sub>) are 3.5 percent H<sub>2</sub>O per 7 ppm CO and 10 percent CO<sub>2</sub> per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent H<sub>2</sub>O per 25 ppm CO and 10 percent CO<sub>2</sub> per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

#### 4. Precision and accuracy.

4.1 *Precision.* The precision of most NDIR analyzers is approximately  $\pm 2$  percent of span.

4.2 *Accuracy.* The accuracy of most NDIR analyzers is approximately  $\pm 5$  percent of span after calibration.

#### 5. Apparatus.

5.1 *Continuous sample* (Figure 10-1).

5.1.1 *Probe.* Stainless steel or sheathed Pyrex<sup>1</sup> glass, equipped with a filter to remove particulate matter.

5.1.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2 *Integrated sample* (Figure 10-2).

5.2.1 *Probe.* Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2.3 *Valve.* Needle valve, or equivalent, to adjust flow rate.

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

5.2.5 *Rate meter.* Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min. (0.035 cfm).

5.2.6 *Flexible bag.* Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft<sup>3</sup>). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

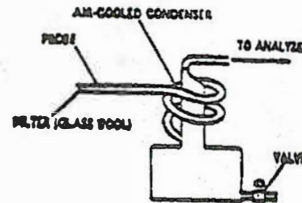


Figure 10-1. Continuous sampling train.

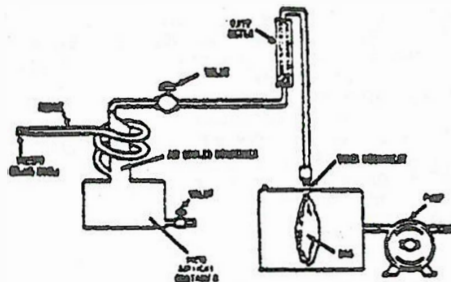


Figure 10-2. Integrated gas sampling train.

5.2.7 *Pitot tube.* Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 *Analysis* (Figure 10-3).

<sup>1</sup> Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

<sup>a</sup> Taken from Federal Register, Protection of Environment, Parts 50-69, page 790-792, July 1, 1975.

5.3.1 *Carbon monoxide analyzer.* Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 *Drying tube.* To contain approximately 200 g of silica gel.

5.3.3 *Calibration gas.* Refer to paragraph 6.1.

5.3.4 *Filter.* As recommended by NDIR manufacturer.

5.3.5 *CO<sub>2</sub> removal tube.* To contain approximately 500 g of ascarite.

5.3.6 *Ice water bath.* For ascarite and silica gel tubes.

5.3.7 *Valve.* Needle valve, or equivalent, to adjust flow rate

5.3.8 *Rate meter.* Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min. (0.035 cfm) through NDIR.

5.3.9 *Recorder (optional).* To provide permanent record of NDIR readings.

6. Reagents.

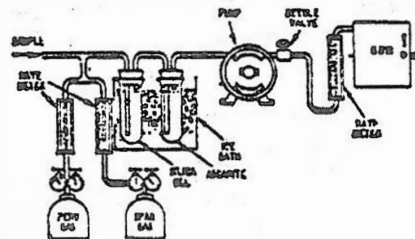


Figure 10-3. Analytical equipment.

6.1 *Calibration gases.* Known concentration of CO in nitrogen (N<sub>2</sub>) for instrument span, prepurified grade of N<sub>2</sub> for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

6.2 *Silica gel.* Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 *Ascarite.* Commercially available.

7. Procedure.

7.1 *Sampling.*

7.1.1 *Continuous sampling.* Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See ¶ 7.2 and 8). CO<sub>2</sub> content of the gas may be determined by using the Method 3 integrated sample procedure (36 FR 24886), or by weighing the ascarite CO<sub>2</sub> removal tube and computing CO<sub>2</sub> concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 *Integrated sampling.* Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 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. CO<sub>2</sub> content of the gas may be determined by using the Method 3 integrated sample procedures (36 FR 24886), or by weighing the ascarite CO<sub>2</sub> removal tube and computing CO<sub>2</sub> concentration from the gas volume sampled and the weight gain of the tube.

7.2 *CO Analysis.* Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in paragraph 8. Purge analyzer with N<sub>2</sub> prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

8. *Calibration.* Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of one hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and CO<sub>2</sub> removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

TABLE 10-1.—Field data

Location _____	Comments: _____
Test _____	
Date _____	
Operator _____	
Clock time	Rotameter setting, liters per minute (cubic feet per minute)

9. *Calculation—Concentration of carbon monoxide.* Calculate the concentration of carbon monoxide in the stack using equation 10-1.

$$C_{CO,stack} = C_{CO,NDIR}(1 - F_{CO_2}) \quad \text{equation 10-1}$$

where:

$C_{CO,stack}$  = concentration of CO in stack, ppm by volume (dry basis).

$C_{CO,NDIR}$  = concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

$F_{CO_2}$  = volume fraction of CO<sub>2</sub> in sample, i.e., percent CO<sub>2</sub> from Orsat analysis divided by 100.

10. *Bibliography.*

10.1 McElroy, Frank, The Intertech NDIR-CO Analyzer, Presented at 11th Methods Conference on Air Pollution, University of California, Berkeley, Calif., April 1, 1970.

10.2 Jacobs, M. B., et al., Continuous Determination of Carbon Monoxide and Hydrocarbons in Air by a Modified Infrared Analyzer, J. Air Pollution Control Association, 9(2):110-114, August 1959.

10.3 MSA LIRA Infrared Gas and Liquid Analyzer Instruction Book, Mine Safety Appliances Co., Technical Products Division, Pittsburgh, Pa.

10.4 Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instruments, Inc., Beckman Instructions 1635-B, Fullerton, Calif., October 1967.

10.5 Continuous CO Monitoring System, Model A5611, Intertech Corp., Princeton, N.J.

10.6 UNOR Infrared Gas Analyzers, Bendix Corp., Roncoveerte, West Virginia.

ADDENDA

A. Performance Specifications for NDIR Carbon Monoxide Analyzers.

Range (minimum).....	0-1000ppm.
Output (minimum).....	0-10mV.
Minimum detectable sensitivity.....	20 ppm.
Rise time, 90 percent (maximum).....	30 seconds.
Fall time, 90 percent (maximum).....	30 seconds.
Zero drift (maximum).....	10% in 8 hours.
Span drift (maximum).....	10% in 8 hours.
Precision (minimum).....	± 2% of full scale.
Noise (maximum).....	± 1% of full scale.
Linearity (maximum deviation).....	2% of full scale.
Interference rejection ratio.....	CO <sub>2</sub> -1000 to 1, H <sub>2</sub> O-500 to 1.

B. Definitions of Performance Specifications.

*Range*—The minimum and maximum measurement limits.

*Output*—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

*Full scale*—The maximum measuring limit for a given range.

*Minimum detectable sensitivity*—The smallest amount of input concentration that can be detected as the concentration approaches zero.

*Accuracy*—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

*Time to 90 percent response*—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

*Rise Time (90 percent)*—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

*Fall Time (90 percent)*—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

*Zero Drift*—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

*Span Drift*—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

*Precision*—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

*Noise*—Spontaneous deviations from a mean output not caused by input concentration changes.

*Linearity*—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

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## 11.0 REFERENCES

1. Collaborative Study of Method 10 - Reference Method for Determination of Carbon Monoxide Emissions from Stationary Sources - Report of Testing. EPA-650/7-75-001. Environmental Protection Agency, Research Triangle Park, N.C. January 1975.
2. McKee, Herbert C., et al. Collaborative Study of Reference Method for the Continuous Measurement of Carbon Monoxide in the Atmosphere (Nondispersive Infrared Spectrometry). Project 01-2811, Contract No. CPA 70-40. Southwest Research Institute, San Antonio, Texas. May 1972.
3. Requirements for Submittal of Implementation Plans and Standards for New Stationary Sources - Emission Monitoring. Federal Register 40, Number 194, October 6, 1975.
4. Part 60 - Standards of Performance for New Stationary Sources - Emission Monitoring Requirements and Revisions to Performance Testing Methods, Federal Register 40, Number 246, December 22, 1975.
5. Part 60 - Standards of Performance for New Stationary Sources - Primary Cooper, Zinc and Lead Smelters, Federal Register 40, Number 10, January 15, 1976, p. 2332-2341.

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Dechant, Richard F., and Peter K. Mueller. Performance of a Continuous NDIR Carbon Monoxide Analyzer. AIHL Report No. 57. Air and Industrial Hygiene Laboratory, Department of Public Health, Berkeley, California. June 1969.

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Smith, Franklin, D.E. Wagoner, and R.P. Donovan. Guidelines for Development of a Quality Assurance Program: Volume VII - Determination of CO Emissions from Stationary Sources by NDIR Spectrometry. EPA-650/ 4-74-005-h. Research Triangle Institute, Research Triangle Park, N.C. for U.S. Environmental Protection Agency, Office of Research and Development, Research Triangle Park, N.C. February 1975.

Smith, Walter S., and D. James Grove. Stack Sampling Nomographs for Field Estimations. Entropy Environmentalists, Inc. Research Triangle Park, N.C. 1973.

Tentative Method of Continuous Analysis for Carbon Monoxide Content of the Atmosphere (Nondispersive Infrared Method). In: Methods of Air Sampling and Analysis. American Public Health Associations, Washington, D.C. 1972.

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## 12.0 DATA FORMS

Blank data forms with identifying titles are included here for the convenience of the handbook user. No page-top documentation is given in the right-hand corners of these forms, as it is on all other pages of this CO method description. Instead to help the user find a corresponding form in the text, a number (Form M10-1.3) is given in the lower right-hand corner to identify the section number (1) and the figure number (3) of the Method 10 (M10) Handbook. Future revisions of this form, if any, will be documented by 1.3A, 1.3B, and so forth. The four in the Method Highlights subsection are shown by the MH following the form numbers below.

<u>Form</u>	<u>Title</u>
1.3	Procurement Log
2.2	Wet Test Meter Calibration Log
2.4A & 2.4B	Rotameter Calibration Data Form (English and Metric Units)
2.8	Calibration Verification Record Chart
3.1 (MH)	Pretest Sampling Checks
3.2 (MH)	Pretest Preparations
4.2	Field Sampling Data Form for CO (Continuous Sample)
4.3	Field Sampling Data Form for CO (Integrated Sample)
4.4 (MH)	On-Site Measurements Checklist
5.1 (MH)	Posttest Sampling Checks
6.1	Carbon Monoxide Calculation Form
8.1	Audit Summary Report
8.2	Method 10 Checklist To Be Used by Auditors

**PROCUREMENT LOG**

Item description	Quantity	Purchase order number	Vendor	Date		Cost	Disposition	Comments
				Ordered	Received			

WET TEST METER CALIBRATION LOG

Wet test meter serial number \_\_\_\_\_ Date \_\_\_\_\_

Wet test meter flow range \_\_\_\_\_ Calibrated by \_\_\_\_\_

Volume of test flask,  $V_s$  \_\_\_\_\_  $\ell$

Satisfactory leak check \_\_\_\_\_

Liquid in wet test meter and reservoir allowed to equilibrate with ambient temperature \_\_\_\_\_

Test number	Manometer reading, <sup>a</sup> mm H <sub>2</sub> O	Final volume ( $V_f$ ), $\ell$	Initial volume ( $V_i$ ), $\ell$	Total volume <sup>b</sup> ( $V_m$ ), $\ell$	Flask volume ( $V_s$ ), $\ell$	Percent error, <sup>c</sup> %

<sup>a</sup>Must be <10 mm (0.4 in.) H<sub>2</sub>O.

<sup>b</sup> $V_m = V_f - V_i$ .

<sup>c</sup>% error =  $100 (V_m - V_s) / V_s =$  \_\_\_\_\_, not to exceed  $\pm 1\%$ .

Signature of calibration person \_\_\_\_\_

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ROTAMETER CALIBRATION DATA FORM (English Units)

Rotameter serial number \_\_\_\_\_ Wet test meter number \_\_\_\_\_  
 Location \_\_\_\_\_ Date \_\_\_\_\_  
 Barometric pressure,  $P_B$  \_\_\_\_\_ in. Hg Calibrated by \_\_\_\_\_

$R_S$ , ft <sup>3</sup> /min	$\theta$ , min	$t_w$ , °F	$D_m$ , in. H <sub>2</sub> O	$V_w$ , ft <sup>3</sup>	$V_r$ , ft <sup>3</sup>	$Q_S$ , ft <sup>3</sup> /min

- $R_S$  = rotameter setting, ft<sup>3</sup>/min (e. g., 0.009, 0.018, 0.027)
- $\theta$  = time of calibration run, min
- $t_w$  = temperature of the gas in wet test meter, °F
- $D_m$  = pressure drop on the wet test meter, in. H<sub>2</sub>O (a negative number if calibrated as in Figure 2.3)
- $V_w$  = gas volume passing through wet test meter, ft<sup>3</sup>
- $V_r$  = gas volume passing through the rotameter corrected to STP, ft<sup>3</sup>
- $Q_S$  = flow rate through rotameter, corrected to STP, ft<sup>3</sup>/min
- $t_s$  = standard temperature, 68°F
- $P_s$  = standard pressure, 29.92 in. Hg
- $V_r = \frac{V_w (P_B + D_m/13.6) (t_s + 460)}{(t_w + 460) P_s} = \frac{17.65 V_w (P_B + D_m/13.6)}{(t_w + 460)} = \text{ft}^3 \text{ at STP.}$
- $Q_S = \frac{V_r}{\theta} = \text{ft}^3/\text{min at STP.}$

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ROTAMETER CALIBRATION DATA FORM (Metric Units)

Rotameter serial number \_\_\_\_\_ Wet test meter number \_\_\_\_\_  
 Location \_\_\_\_\_ Date \_\_\_\_\_  
 Barometric pressure,  $P_B$  \_\_\_\_\_ mm Hg Calibrated by \_\_\_\_\_

$R_S$ , ℓ/min	$\theta$ , min	$t_w$ , °C	$D_m$ , mm H <sub>2</sub> O	$V_w$ , ℓ	$V_r$ , ℓ	$Q_S$ , ℓ/min

$R_S$  = rotameter setting, ℓ/min (e.g., 0, 0.50, 0.75)

$\theta$  = time of calibration run, min

$t_w$  = temperature of the gas in the test meter, °C

$D_m$  = pressure drop on the wet test meter, mm H<sub>2</sub>O (a negative number if calibrated as in Figure 2.3)

$V_w$  = gas volume passing through wet test meter, ℓ

$V_r$  = gas volume passing through the rotameter corrected to STP, ℓ

$Q_S$  = flow rate through rotameter, corrected to STP, ℓ/min

$t_s$  = standard temperature, 20°C

$P_s$  = standard pressure, 760 mm Hg

$$V_r = \frac{V_w (P_B + D_m/13.6) (t_s + 273)}{(t_w + 273) P_s} = \frac{0.386 V_w (P_B + D_m/13.6)}{(t_w + 273)} = \ell \text{ at STP.}$$

$$Q_S = \frac{V_r}{\theta} = \ell/\text{min at STP.}$$







CARBON MONOXIDE CALCULATION FORM

Plant \_\_\_\_\_ Date \_\_\_\_\_

Sample location \_\_\_\_\_

Test number \_\_\_\_\_

Bag number \_\_\_\_\_

Operator \_\_\_\_\_

CO Concentration

$$C_{CO_{NDIR}} = \_ \_ \_ \text{ ppm (dry basis).}$$

$$F_{CO_2} = \_ \_ \_ \% \div 100 = \underline{0. \_ \_ \_}$$

$$C_{CO_{stack}} = C_{CO_{NDIR}} (1 - F_{CO_2}).$$

$$= \_ \_ \_ (1 - \underline{0. \_ \_ \_}) = \_ \_ \_ \text{ ppm (dry basis).}$$

Errors:

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# AUDIT SUMMARY REPORT

Tester \_\_\_\_\_ Source \_\_\_\_\_

Analyzer \_\_\_\_\_ Range \_\_\_\_\_

Analyzer serial number \_\_\_\_\_

Auditor \_\_\_\_\_ Date \_\_\_\_\_

Cylinder ID \_\_\_\_\_ Concentration \_\_\_\_\_

Remarks \_\_\_\_\_

\_\_\_\_\_  
 \_\_\_\_\_

Audit cylinder conc $(C_{CO})_a$ , ppm	Analyzer response $(C_{CO})_m$ , ppm	Percent accuracy, $(\%A)$ , %*

$$*\%A = \frac{(C_{CO})_m - (C_{CO})_a}{(C_{CO})_a} \times 100.$$

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METHOD 10 CHECKLIST TO BE USED BY AUDITORS

			OPERATION
yes	no	comment	
			<u>Presampling Preparations</u>
—	—	—	1. Knowledge of process conditions
—	—	—	2. Traceability of calibration gas established
—	—	—	3. Calibration of pertinent equipment, in particular, the NDIR
			<u>On-Site Measurements</u>
—	—	—	4. Leak test of sampling train
—	—	—	5. NDIR warmup per manufacturer's recommendations
—	—	—	6. Purging the train prior to sampling
—	—	—	7. Proportional sampling
—	—	—	8. Frequency of zero and span checks
—	—	—	9. Drying agents checked and replaced frequently
			<u>Postsampling</u>
—	—	—	10. Transfer and handling of sample
—	—	—	11. Data reduction procedure/check
—	—	—	12. Calibration checks
GENERAL COMMENTS:			

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