SOURCE TEST METHODS WITH CALCULATIONS

METHOD 100
TEST PROCEDURES FOR THE DETERMINATION OF NITROGEN OXIDES, CARBON MONOXIDE AND DILUENT GASES BY CONTINUOUS EMISSION MONITORING

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1. Principle and Applicability

1.1 Principle

A stack gas sample is extracted continuously from the sampling port and conditioned to remove water and particulate matter before analysis by instrumental analyzers. Nitrogen oxide (NO_x) emissions are analyzed by chemiluminescence. Carbon monoxide and carbon dioxide emissions are analyzed with non-dispersive infrared radiation. Oxygen is analyzed by an electrochemical cell or by a paramagnetic detector.

1.2 Applicability

This method is used to quantify emissions of nitrogen oxides, carbon monoxide, carbon dioxide and oxygen from stationary sources. Generally, this method is used on turbines, internal combustion engines, boilers and other combustion sources.

2. Range and Sensitivity

2.1 Analytical Range

The analytical range is determined by the instrument design. For this method, a portion of the analytical range is selected by choosing the span of the monitoring system. The span of the monitoring system shall be selected such that the raw pollutant gas concentration equivalent to the emission standard is not less than 25 percent of the span. If at any time during a run the measured gas concentration exceeds the span, the run shall be considered invalid for the determination of compliance.

2.2. Sensitivity

The minimum detectable limit depends on the analytical range, span, and signal-to-noise ratio of the measurement system. For a well designed system, the minimum detectable limit should be less than 2 percent of the span.

3. Definitions

3.1 Measurement System

The total equipment required for the determination of gas concentration. The measurement system consists of the following major subsystems:

3.1.1 **Sample interface:** That portion of a system used for one or more of the following: sample acquisition (probe), sample transport, sample conditioning (condenser), or protection of the analyzers from the effects of the stack effluent (filter).

3.1.2 **Gas analyzer:** That portion of the system that senses the gas to be measured and generates an output proportional to its concentration.

3.1.2.a Nitrogen oxides (NO_x) analyzer: This instrument detects NO_x and produces an electronic signal proportional to its concentration. An analyzer based on the principles of chemiluminescence, to continuously determine the NO_x concentration in the sample gas stream, is acceptable.

3.1.2.b Nitrogen dioxide (NO_2) to nitric oxide (NO) converter: This component within the NO_x analyzer converts the NO_2 of the NO_x gases to NO for subsequent analysis by the NO_x analyzer. The converter must be at least 90 percent efficient. A low temperature converter shall be used for stack effluent containing ammonia (NH_3).

3.1.2.c Carbon monoxide (CO) analyzer: This instrument detects CO and produces an electronic signal proportional to its concentration. A Luft-type non-dispersive infrared analyzer (NDIR) or equivalent is acceptable.

3.1.2.d Oxygen (O_2) analyzer: This instrument detects O_2 and produces an electronic signal proportional to its concentration. An electrochemical (fuel cell) type analyzer or paramagnetic type analyzer is acceptable.

3.1.2.e Carbon dioxide (CO_2) analyzer (optional): This instrument detects CO_2 and produces an electronic signal proportional to its concentration. A non-dispersive infrared (NDIR) analyzer or its equivalent is acceptable.

3.1.2.f Standard gas dilution system: A standard gas dilution system provides known values of calibration gases through controlled dilution of high level calibration gases with an appropriate dilution gas. A standard gas dilution system may be a capillary system, an orifice system or a set of mass flow controllers.

3.1.3 **Data acquisition:** A strip chart recorder, analog computer, or digital recorder for recording measurement data from the analyzer output. Recorders shall employ strip chart paper with 100 divisions minimum.

3.2 Test Duration

A compliance test consists of three 30 or 40 minute subtests, depending on the type of site equipment. The duration of testing may vary for specific applications with written District approval. Test results demonstrate whether site emissions meet or exceed Permit to Operate limits. The Compliance Division determines if an exceedance is a violation of the Permit to Operate.

3.3 Span

The specified upper limit of a gas concentration measurement range for source categories in the applicable regulations. This is the full scale value of the data recorder in the same units for each particular gas.

3.4 Calibration Gases

An assayed gas of known concentration in an appropriate diluent gas. EPA Protocol 1 gases will be utilized to calibrate NO_x, CO, CO_2 and O_2 instrumentation.

3.5 Analyzer Calibration

The value of the gas concentration exhibited by the gas analyzer when the calibration gas is introduced directly to the analyzer.

3.6 System Calibration

The value of the gas concentration exhibited by the gas analyzer when the calibration gas is introduced at the probe.

3.7 Analyzer Calibration Error

The difference between the gas concentration exhibited by the gas analyzer and the known concentration of the calibration gas when the calibration gas is introduced directly to the analyzer (analyzer calibration).

3.8 Standard Gas Dilution System: Field Evaluation

If a standard gas dilution system is to be used for obtaining high, mid or low-range calibration gases, a field evaluation of the dilution system must be performed once per day. 40 CFR 51, Appendix M, Method 205 contains the protocol for this evaluation.

3.9 Multi-Point Calibration Check

Maximum deviation, as a percent of span, between the actual mid-range calibration gas analyzer response and the reading predicted by a straight line drawn between the high-range and zero analyzer calibration gas readings.

3.10 NO_2 Converter Efficiency Check (On-Site)

An NO_2 converter efficiency check is performed on-site, prior to testing, for high NO_2 emission sites. An NIST traceable NO_2 cylinder gas may be used. The NO_2 gas is sampled in both the total NO_x mode and the NO mode. The difference between the two responses is then compared to the NO_2 cylinder concentration value.

3.11 Sampling System Bias

The difference between the gas concentration exhibited by the measurement system when a known concentration gas is introduced at the sampling probe (system calibration) and when the same gas is introduced directly to the analyzer (analyzer calibration).

3.12 Analyzer Calibration Drift

The difference in the analyzer responses at the zero, high, mid or low-concentration level during the initial and final analyzer calibration checks for a 30 or 40 minute subtest. During this period, there shall be no unscheduled maintenance, repair, or adjustments to the measurement system. This difference includes the electronic drift of the instrument and the recorder.

3.13 System Calibration Drift

The difference in the analyzer responses at a zero, mid or high-range concentration level during the initial and final system calibration checks for a 30 or 40 minute subtest. During this period there shall be no unscheduled maintenance, repair, or adjustments to the measurement system. This difference includes the electronic drift of the instrument and the recorder.

3.14 Response Time

The amount of time required for the measurement system to display 95 percent of a step change in the gas concentration on the data recorder.

3.15 Interference Response

The response of the measurement system to a component in the sample gas, other than the gas component being measured. During instrument review for acceptance, interferences must be evaluated. Manufacturer's data may be used for all or part of the interference response testing. Field tests may be necessary for some sites.

3.16 Linearity

A multi point calibration consisting of four calibration gases and a zero. A determination is made for the deviation between readings for each of the calibration values (predicted from a straight line between the high and zero calibrations) and the actual analyzer response.

3.17 Laboratory Converter Efficiency

A procedure outlined in 40 CFR 60, Appendix A, Method 20 for the determination of the NO_2 converter efficiency. A Tedlar bag is filled with a standard gas of NO in N_2 and diluted 1:1 with zero air. The gas is then sampled through the converter and the initial and final NO_x values determined to yield the converter efficiency.

3.18 Stratification

As determined by traverse point sampling, stratification is a variance in the O_2, NO_x or CO concentration greater than 10 percent from the average concentration.

3.19 Calibration Curve

A graph or other systematic method of establishing the relationship between the analyzer response and the actual calibration gas concentration introduced to the analyzer.

3.20 Stable Value

A stable value of a calibration gas is equivalent to a change of less than ± 1 percent of the span for 30 seconds or less than ± 5 percent of the measured average concentration for two minutes. All calibration gas responses must be recorded until stable values are obtained.

3.21 High NO_2 Emission Site

A site with a stack effluent that contains greater than 5 percent of the total NO_x as NO_2.

4. Measurement System Performance Specifications

4.1 Range and Sensitivity

The minimum and maximum measurable concentrations of NO_x, CO, CO_2 and O_2 depend on the specific analyzers. The minimum sensitivity of the analyzer will be ± 2 percent of the span. To test for compliance with a specific emission limit, select an analyzer span that puts this limit within 25 - 80 percent of the span.

4.2 Calibration Gas

4.2.1 **Certification:** The NO, CO, CO_2 and O_2 calibration gases must be certified by EPA Revised Traceability Protocol Number 1, "Procedure for NBS-Traceable Certification of Compressed Gas Working Standards Used for Calibration and Audit of Continuous Source Emission Monitors".

4.2.2 **Concentration:** Use three calibration gases as specified below:

4.2.2.a High-range gas: The concentration should be equivalent to 80 - 100 percent of the span.

4.2.2.b Mid-range gas: The concentration should be equivalent to 40 - 60 percent of the span.

4.2.2.c Zero gas: The impurity concentration should be less than 1 percent of the span. Purified ambient air may be used for the zero gas by passing compressed air through a heatless dryer, a catalytic oxidizer and a carbon dioxide scrubber. Use ultra zero grade nitrogen gas for zeroing the O_2 analyzer.

4.2.3 **Gas super blends:** Super blends, i.e. gas mixtures of nitric oxide, carbon monoxide and carbon dioxide with nitrogen as the balance gas, may be used for simultaneous calibration of multiple analyzers. The true concentration of each component must be confirmed using NIST traceable standards at least every 12 months.

4.2.4 **Standard gas specifications for calibration gases:**

4.2.4.a Nitric oxide must be oxygen free (less than 10 ppm O_2) and balanced with nitrogen.

4.2.4.b Nitrogen dioxide must be balanced with nitrogen or ultra-pure air and be NIST traceable.

4.2.4.c Carbon monoxide and carbon dioxide must be balanced with nitrogen or ultra-pure air.

4.24.d Oxygen must be balanced with nitrogen.

4.2.4.e Minimum cylinder pressure: No compressed gas cylinder standard may be used when its gas pressure is less than 300 psi as indicated by the cylinder pressure gauge.

4.2.4.f NO, CO, CO_2 and O_2 must be EPA Protocol 1 certified gases with certificates of analysis.

4.2.4.g Either a standard gas dilution system or a second calibration gas must be used to obtain the mid-level calibration gas in order to perform a multi-point calibration for each analyzer. Both the diluted and undiluted calibration gases shall be analyzed without changing the instrument potentiometers (this usually is equivalent to using the same span setting).

4.2.5 **Zero calibration gas:** The zero calibration gas exhibits a zero response on the analyzers. Acceptable gases include:

4.2.5 a For NO/NO_x: Nitrogen, ambient air or a gas certified to contain less than 1 ppm of pollutant.

4.2.5 b For CO/CO_2: Nitrogen, ambient air.

4.2.5 c For O_2: Nitrogen, NO_x or CO in nitrogen.

4.3 Analyzer Calibration Error

Less than ± 2 percent of the span for the zero, mid-range and high-range calibration gases.

4.4 Standard Gas Dilution System: Field Evaluation

The protocol for the standard gas dilution system field evaluation is located in 40 CFR 51, Appendix M, Method 205, "Verification of Gas Dilution Systems for Field Instrument Calibrations."

The average instrument response for each triplicate injection at each dilution level shall not differ by more than ± 2 percent from any single injection for that dilution level. For each dilution level, the average of the analyzer responses shall be within ± 2 percent of the predicted concentration. The average of the analyzer responses for the mid-range calibration gases injected directly to the analyzer (bypassing the standard gas dilution system) shall be within ± 2 percent of the certified cylinder concentration.

4.5 Multi-Point Calibration Check

The maximum deviation between the actual mid-range calibration gas analyzer response and the reading, predicted by a straight line drawn between the high-range and zero calibration gas readings, shall be less than ± 2 percent of the span.

4.6 NO_2 Converter Efficiency Check (On-site)

For high NO_2 emission sites, a NO_2 converter efficiency check must be performed prior to each test. A NO_2 converter efficiency of greater than 90 percent is required.

4.7 Sampling System Bias

For NO, CO, CO_2 and O_2, a sampling system bias of less than ± 5 percent of the high or mid-range calibration gas when introduced directly into the analyzer, is acceptable. The zero calibration gas sampling system bias shall be less than ± 2 percent of the span. For high NO_2 emission sites, the sampling system bias for NO_2 shall be less than ± 15 percent of the NO_2 calibration gas when introduced directly into the analyzer (in NO_x mode).

4.8 Analyzer Calibration Drift

For NO, NO_2, CO, CO_2 and O_2, an analyzer calibration drift of less than ± 2 percent of the span over the period of each subtest is acceptable.

4.9 System Calibration Drift

For NO, NO_2, CO, CO_2 and O_2, a drift of less than ± 2 percent of the span over the period of each subtest is acceptable.

For a system calibration drift of greater than ± 2 percent of the span, a sampling system bias check must be performed before any adjustments are made to the analyzer. If an adjustment is made to the analyzer to correct for the analyzer calibration error, a subsequent system calibration shall be performed to check that sampling system bias requirements have been met.

4.10 Correction Factors in Computations

Any instrument should be reset after each subtest when its drift exceeds the limit. When drift limits are exceeded, the starting and ending subtest values for the calibration and zero gases should be used to calculate the stack emission values.

4.11 Interference Response

Conduct an interference response test of each analyzer prior to its initial use in the field. Thereafter, recheck the measurement system if changes in the instrumentation could alter the interference response. In lieu of conducting the interference response test, instrument vendor data can be submitted for District approval. Interference response should be less than ± 2 percent of the span value.

4.12 Linearity

Perform a multi-point calibration of four points plus zero gas. Stable values at each level are required. Perform a linear regression using the highest gas reading and the zero gas reading. Use the slope and intercept of this regression line to calculate the predicted concentration at each of the three mid-levels. If for each such level, the calibration concentration reading is within ± 2 percent of the value predicted by the ideal line, the instrument is linear. A linearity check must be performed semi-annually. For nonlinear instruments, appropriate adjustments should be made.

4.13 Laboratory Converter Efficiency

Add gas from a calibration gas cylinder of NO in N_2 to a clean, evacuated, leak-tight Tedlar bag. Dilute this gas approximately 1:1 with zero air. Immediately attach the bag outlet to the calibration valve assembly and begin operating the sampling system. Operate the sampling system, recording the NO_x response for at least 30 minutes. To determine the converter efficiency, compare the final NO_x value to the initial NO_x value. If the response at the end of 30 minutes decreases by more than 2 percent of the highest peak value, the converter is unacceptable and appropriate repairs must be made. This converter efficiency must be performed every six months or when the converter efficiency check (see Section 3.10) is below 90 percent. Consultant test reports require documentation. Alternative procedures may be used with prior District approval.

5. Apparatus

5.1 Measurement System

Any measurement system for NO, NO_2, CO, CO_2 and O_2 that meets the specifications of this method. A schematic of an acceptable measurement system is shown in Figure 100-1, Sampling System. The essential components of the measurement system are:

5.1.1 **Sample probe:** Use a stainless steel, glass or equivalent open straight tube of sufficient length to traverse the sample points. Heat it if necessary to prevent condensation.

5.1.2 **Sample line:** If it is necessary to prevent condensation, heat the sample tubing to at least 95°C. Because of the reactivity of NO_x, stainless steel or Teflon tubing must be used to transport the stack gas to the sample conditioners and analyzers. Systems using materials other than Teflon, glass or stainless steel must have District approval prior to testing.

5.1.3 **Sample line vacuum gauge:** During system calibrations through the probe, the tester shall demonstrate that the sample line from the probe to the sample pump is not pressurized due to the introduction of the calibration gas. A sample line vacuum gauge is an acceptable method for this check.

5.1.4 **Moisture removal system:** A moisture removal system that is at least as efficient as an ice bath must be utilized. It must not remove CO, NO, NO_2 or O_2.

For high NO_2 emission sites, an NO_2 recovery measurement must demonstrate that the condensed water does not absorb the NO_2 gas. An impinger system with dry ice in a 50/50 mixture of water and ethylene glycol is one such acceptable system (see Figure 100-1, Sampling System). Alternative systems must be demonstrated and approved by the District prior to use.

5.1.5 **Particulate filter:** An out-of-stack glass-fiber filter of the type specified in 40 CFR 60, Appendix A, Method 5 (0.3 micron, 90 percent efficient) is required. A relatively clean filter is required during testing.

5.1.6 **Sample pump:** A non-reactive, leak-free pump to transport the sample gas through the system at a flow rate sufficient to minimize transport delay is required. The pump must be stainless steel lined, Teflon lined or the equivalent.

5.1.7 **Sample gas manifold:** A manifold to divert a portion of the sample gas stream to the analyzers is required. The manifold must be constructed of non-reactive material such as glass, Teflon, stainless steel or the equivalent.

5.1.8 **Pressure regulator:** A pressure regulator on the sample gas manifold must be used to assure that stack gases and standard gases are delivered to the instruments at the same pressure.

5.1.9 **NO_2 converter:** An NO_2 converter is required for all high NO_2 emission sites.

When NH_3 is also a constituent of the stack gas, choose a NO_2 converter that prevents interference by the NH_3 on the NO_x readings. A low temperature molybdenum converter is acceptable.

5.1.10 **Dilution system for calibration gases:** A standard gas dilution system may be used to dilute standard certified calibration gases with standard dilution gases in field conditions. The standard gas dilution system must be operated according to the manufacturer's instructions and meet the specifications for the field evaluation of standard gas dilution systems (see Section 4.4).

6. Test Procedures

6.1 Pre-test Procedures (off-site)

6.1.1 **Site survey:** If necessary, contact the site to arrange for a site survey to collect the data, including verifying ports, access to them and electrical supply lines for instruments (shown in Figure 100-2, Site Survey Data). This information is recorded in the Site Profile files.

6.1.2 **Port location:** The port hole should be of sufficient diameter to allow complete entry of the probe for a stack traverse. If possible, sampling should be conducted at a location where there is no stratification. Ports should be at least two diameters or five feet (whichever is less) upstream from the end of the stack and meet the 2 and 1/2 diameter criteria of 40 CFR 60, Appendix A, Method 1. Alternative port locations require prior District approval.

6.1.3 **Instrument response time:** Record instrument response time during five-point linearity checks every six months. A response time of less than 30 seconds is recommended.

6.1.4 **Linearity:** Perform semi-annual linearity checks (Section 4.12). Documentation shall be kept on file.

6.1.5 **Amount of NO_2 in the exhaust gas:** Testing procedures are, in part, determined by the amount of NO_2 in the exhaust gas. For high NO_2 emission sites, perform the following procedures before arriving on site:

6.1.5.a Evaluation by the District's engineering division will be used to determine whether NO_2 in the exhaust gas is expected to be greater than 5 percent of the total NO_x in the exhaust gas. On-site evaluation may be required at sites where there is insufficient engineering information.

6.1.5.b A moisture removal system that does not remove NO_2 in moist stack gas must be used (see Section 5.1.4).

6.1.5.c The sampling time at high NO_2 sites must be at least 40 minutes per subtest: 30 minutes of testing using the NO_2 converter (NO_x mode) and at least 10 minutes of testing performed bypassing the converter (NO mode). An analyzer that performs both the NO_x and NO analysis concurrently, may test for only the 30 minute period, obtaining averaged values for NO_x and NO over that time period.

6.1.5.d NO_2 sampling system bias checks and on-site NO_2 converter efficiency checks are required at high NO_2 emission sites. Therefore, an NIST traceable NO_2 gas cylinder will be needed on-site.

6.2 Pre-test Procedures (On-Site)

6.2.1 **Assembly:** Assemble the sampling system and analyzers as shown in Figure 100-1, Sampling System.

6.2.2 **Documentation:** Record data on a chart recorder as shown in Figure 100-3, Strip Chart Data (Tests Performed by APCD), and Figure 100-4, Standard Chart Notations. Record any unusual events that occur during the testing period.

6.2.3 **Warm-up:** Warm up the instruments according to the manufacturer's instructions. Zero the chart recorder by shunting the input electrical signal.

6.2.4 **Vacuum leak check:** Plug the probe and establish a vacuum using the same vacuum pressure to be used during normal sampling. Sample line rotameters giving zero readings demonstrate a leak-free system.

6.2.5 **Zero instruments:** Introduce zero air into the analyzers and zero the instruments according to the manufacturer's instructions. Set the flow and back pressure gauges and record the settings.

6.2.6 **Standard gas dilution system: field evaluation:** If a standard gas dilution system is to be used for high, mid or low-range calibration values, perform a field evaluation of the standard gas dilution system as outlined in 40 CFR 51, Appendix M, Method 205. The evaluation need only be performed once per day using an analyzer chosen by the tester.

6.2.7 **Multi-point calibration check:** An on-site multi-point analyzer calibration that uses zero, mid-range and high-range gases must be performed. The maximum deviation between the actual mid-range calibration gas analyzer response and the reading predicted by a straight line drawn between the high-range and zero calibration gas readings shall be less than $\pm$ 2 percent of the span.

6.2.8 **NO_2 converter efficiency check (On-site):** For high NO_2 emission sites, an on-site NO_2 converter efficiency check must be performed before stack testing. This converter efficiency check is performed with NO_2 gas in a compressed gas cylinder. The NO_2 gas is sampled in both the total NO_x mode and the NO mode. Stable responses in both modes are necessary. The NO_2 value is the difference between the two responses. This value is compared to the cylinder concentration value. The NO_2 converter efficiency must be greater than 90 percent.

6.2.9 **Sampling system bias:** Conduct a sampling system bias check using NO, NO_2 (if necessary), CO, CO_2 and O_2 zero and mid- or high-range calibration gases. For the mid- or high-range calibration gas, use the gas that is closest to the stack emission value. The sampling system bias check consists of performing an analyzer calibration and comparing that analyzer response to the response for the system calibration when using the same zero or mid- or high-range calibration gas. The system calibration gas must be introduced at the probe or at the connection of the probe to the sample line tubing. When the system calibration gas is introduced at the probe or at the outlet of the probe, it must be demonstrated that the calibration gas is not pressurizing the sampling system. The sampling system bias check performance specifications listed in Section 4.7 must be met. Stable values are required.

Values outside the limits indicate a system which is not working properly and must be corrected before testing proceeds. Once the sampling system bias checks have been performed, do not make any adjustments to the analyzer calibrations or the sampling system prior to testing.

6.2.10 **Stratification:** At the District's option, the determination of stratification may be omitted for renewal testing if non-stratification test data are available and no modifications have been made to the stack. If one of these two criteria are not met, a stratification check must be performed. Select traverse points from criteria in 40 CFR 60, Appendix A, Method 1 for non-particulate traverses. For stacks less than twelve inches in diameter select eight traverse points and use the stack percentage criteria from Method 1. For gas turbines follow criteria in 40 CFR 60, Appendix A, Method 20 for the selection and sampling of traverse points. Stratification testing should be performed during periods of steady operation.

Perform the stratification check at each of the chosen probe points. At each point, compute time-averaged values. Select the maximum and minimum values of those results. If this maximum or minimum differs from the average concentration of the stack by more than $\pm$ 10 percent, stratification exists. Stratification can be measured for either pollutant gases (NO_x or CO) or diluent gases (O_2 and CO_2) in units of concentration. If stratification does not exist, sample at a point near the center of the stack. Data from the stratification check may be used for at least part of the first subtest.

6.2.11 **Sampling system response time:** The sample train response time may be measured simultaneously with the system calibration check. The response time corresponds to an instrument response of 95 percent of the final value. (A 90 second maximum is recommended.)

6.3 Testing

6.3.1 **Low NO_2 emissions:** For low NO_2 emission sites, a single test consists of three 30 minute subtests with zeros and high or mid-range system calibrations introduced at the probe for NO, O_2, CO and CO_2 before and after each subtest. Sample system bias checks are performed prior to the first subtest and after the third subtest.

6.3.2 **High NO_2 emissions:** For high NO_2 emission sites, a single test must consist of three subtests of at least 40 minutes in duration. Thirty minutes of each subtest will be performed with the analyzer in the NO_x sampling mode followed by at least 10 minutes of sampling in the NO mode. For analyzers that concurrently measure NO_x and NO, three 30

minute subtests are acceptable. Zero and mid or high-range system calibrations are required for NO, NO_2, CO, CO_2 and O_2 as well as the direct measurement of analyzer calibration error for NO and NO_2 between each subtest.

6.3.3 **Sampling:** Introduce sample gas to the analyzer at the same flow rate and pressure used to calibrate the analyzers. Record these values.

6.3.4 **Moisture removal:** Ensure that the moisture removal system is effective throughout the test. A thermocouple, dial thermometer or visual check of the ice or dry ice bath may be used.

6.3.5 **System calibration drift, analyzer calibration drift:** After each subtest, perform zero and mid or high-range system calibration checks for NO, (NO_2 if necessary), CO, CO_2 and O_2 before any adjustments to the instruments are made. For high NO_2 emission sites; perform the analyzer calibration checks for NO and NO_2 in addition to the system calibration checks. Stable values are required. Record the data on the chart.

If the system calibration drift for the zero or mid or high-range calibration gas is outside the allowed limits, perform the analyzer calibration error check (to determine the sampling system bias and analyzer calibration drift). Readjust the analyzers if necessary. Do not make any analyzer or system adjustments prior to the analyzer calibration error check. Perform a sampling system bias check before starting the next subtest. If no adjustment is necessary for a subsequent subtest, continue testing. Annotate any adjustments on the chart. For each subtest, the subtest is acceptable if the average of the initial and final sampling system bias checks meets the performance specifications listed in Section 4.7.

6.3.5.a Alternative: An alternative to conducting NO and NO_2 sampling system bias checks before and after each subtest is to conduct NO analyzer calibration error checks before and after each subtest and conduct NO and NO_2 sampling system bias checks only before the first subtest and after the third subtest. The average of the pre-test and post-test NO and NO_2 sampling system bias results shall be used to correct the NO and NO_2 portions of the stack gas averaged over the three subtests. However, final sampling system bias results that are greater than $\pm$ 5 percent for NO, or greater than $\pm$ 15 percent for NO_2, are not acceptable and all three subtests will be considered invalid.

For CO, O_2 and CO_2, sampling system bias checks may be performed just once prior to the first subtest and after the third subtest. CO, O_2 and CO_2 analyzer calibration error checks must be performed after each subtest and used to correct the emission values for drift and analyzer calibration error. The average of the initial and final sampling system bias checks are used to correct the CO, O_2 and CO_2 values for system bias. However, a final sampling system bias check of greater than $\pm$ 5 percent is not acceptable and will invalidate all three subtests.

6.4 Post-test Procedures

6.4.1 **Sampling system bias:** After the last subtest, perform the same sampling system bias check with the calibration gases used at the start of the testing. Introduce zero and high or mid-range calibrations gases at the probe and compare these responses to those obtained when the same zero and high or mid-range gases are introduced directly to the analyzer. A stable value is required. The sampling system bias check performance specifications listed in Section 4.7 must be met.

If, for any subtest, the initial NO_2 sampling system bias is less than 15 percent but the final sampling system bias is greater than 15 percent and the average bias value is equal to or less than 15 percent, the test will be acceptable. If the average is greater than 15 percent, the subtest will not be acceptable and the subtest shall be repeated once an acceptable sampling system bias is achieved.

Alternatively, for NO, CO, O_2, CO_2 and NO_2 sampling system bias checks performed only at the beginning of the first subtest and the end of the third subtest (see Section 6.3.5.a), for any test where the final NO, CO, CO_2 or O_2 sampling system bias is greater than 5 percent and/or the final NO_2 sampling system bias is greater than 15 percent, the test (three subtests) shall be considered invalid.

7. Calculations

7.1 Chart Data Reduction

7.1.1 **From the chart data,** the average values of each subtest for (NO_x, CO, O_2, and CO_2 and NO if applicable) are recorded.

7.1.2 **Alternatively, a data logger** may be used to record data.

7.1.3 **Stratification testing** requires averaging of the individual points of the traverse for each subtest.

7.2 Emission Calculations

7.2.1 **Average concentrations:** Determine the time-averaged concentrations of NO, NO_x, CO, O_2 and CO_2 on a dry basis for each subtest from the chart recordings. The time averaging should be calculated for each sampling point or at least every one minute, whichever is relevant, for each subtest.

7.2.2 **Effluent gas concentrations - Low NO_2 emission sites:** (stack gas effluent which contains less than 5 percent of the total NO_x as NO_2). Calculate the NO_x, O_2, CO and CO_2 effluent gas concentrations, corrected for analyzer drift, calibration error and sampling system bias, using the following equation. Calculate the effluent gas concentration for each subtest. Average the three subtest values for an overall test average.

$$C_{gas} = (C_{avg} - C_o) \frac{C_{ma}}{(C_m - C_o)}$$

Where:

C_{gas} = Effluent gas concentration, dry basis, ppm.

C_{avg} = Average gas concentration indicated by the analyzer, in Section 7.2.1, dry basis, ppm.

C_{ma} = Actual certified concentration of the mid or high-range calibration gas used for the system calibration checks, ppm.

C_m = Average of the initial and final system calibration responses for the mid- or high-range calibration gas, ppm.

C_o = Average of the initial and final system calibration responses for the zero calibration gas, ppm.

7.2.3 **Effluent gas concentrations - High NO_2 emission sites:** (stack effluent which contains more than 5 percent of the total NO_x as NO_2). Calculate the CO, O_2 and CO_2 effluent gas concentrations as specified in Section 7.2.2.

Calculate the averaged NO_x, NO and NO_2 effluent gas concentrations, for each subtest, corrected for analyzer drift, calibration error and sampling system bias, using the following formulas:

$$NO_{gas} = (NO_{avg} - NO_o) \frac{(NO_{ma})}{(NO_{md} - NO_{od})} \frac{(NO_{md} - NO_{od})}{(NO_m - NO_o)}$$

$$NO_{2gas} = [(NO_{xavg} - NO_{od}) - (NO_{avg} - NO_{od})] \frac{(NO_{ma})}{(NO_{md} - NO_{od})} \frac{(NO_{2md} - NO_{od})}{(NO_{2m} - NO_{od})}$$

$$NO_{xgas} = NO_{gas} + NO_{2gas}$$

Where:

NO_{gas} = The NO concentration in the stack effluent, dry basis, ppm.

NO_{avg} = The average NO concentration indicated by the analyzer, Section 7.2.1, dry basis, ppm.

NO_{od} = The average of the initial and final analyzer calibration responses for the zero NO calibration gas, ppm.

NO_{ma} = The actual certified concentration of the mid- or high-range NO calibration gas, ppm.

NO_{md} = The average of the initial and final analyzer calibration responses for the mid or high-range NO calibration gas, ppm.

NO_m = The average of the initial and final system calibration responses for the mid- or high-range NO calibration gas, ppm.

NO_o = The average of the initial and final system calibration responses for the zero NO calibration gas, ppm.

NO_{2gas} = The NO_2 concentration in the stack effluent, dry basis, ppm.

NO_{xavg} = The average NO_x concentration indicated by the analyzer, Section 7.2.1, dry basis, ppm.

NO_{2md} = The average of the initial and final analyzer calibration responses for the NO_2 calibration gas, ppm.

NO_{2m} = The average of the initial and final system calibration responses for the NO_2 calibration gas, ppm.

NO_{xgas} = The total NO_x concentration in the stack effluent, dry basis, ppm.

For the alternative test procedures outlined in section 6.3.5.a, use the formula in section 7.2.2 to correct the emission values (NO_x, CO, O_2, CO_2) for calibration error and drift where the zero and mid- or high-range calibration responses are those taken directly from the analyzer. For high NO_2 emissions, use the NO zero and mid- or high-range calibration responses directly from the analyzer to correct both NO_x and NO for calibration error and drift. Calculate the NO_2 values for each subtest. Use the average values of the beginning and ending sample system bias checks to correct the emission data for the system bias. To calculate total corrected NO_x, add the corrected NO and NO_2 values.

7.2.4 **NOx and CO gas concentrations corrected to x% O_2:** For specified emissions limits corrected to x% O_2, use the following equation:

$$C_{gas}\ @\ x\%\ O_2 = C_{gas}\ \frac{(20.95 - x\%\ O_2)}{(20.95 - C_{gas\ O2\%})}$$

Where

$C_{gas}\ @\ x\%\ O_2$ = The stack effluent concentration, for either NO_x or CO, corrected to x% O_2, ppm.

C_{gas} = The stack gas effluent concentration, for either NO_x or CO, determined in Section 7.2.2 or 7.2.3., dry basis, ppm.

$C_{gas\ O2\%}$ = Stack effluent concentration of O_2, determined in Section 7.2.2 or 7.2.3., dry basis, %.

8. Report

8.1 Report Folder

The report folder includes the sections shown in Figure 100-3, Strip Chart Data (Tests Performed by APCD) and Figure 100-4, Standard Chart Notations. The report can be completed using a computer program.

8.2 Audit Check

An independent audit check is necessary for all reports.

8.3 Completed Report

A completed report includes all supporting test documentation, source test report and results and an engineering report specifying operating parameters including those listed in the Permit to Operate conditions.

9. References

1. Code of Federal Regulations, Title 40, Part 51, "Verification of Gas Dilution Systems for Field Instrument Calibrations", Appendix M, Test Method 205, 1995.

2. Code of Federal Regulations, Title 40, Part 60, "Standards of Performance for New Stationary Sources", Appendix A, Test Methods 1, 5 and 20, 1994.

3. Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III. Stationary Sources Specific Methods. Section 3.0.4., "Procedure for NBS-Traceable Certification of Compressed Gas Working Standards Used for Calibration and Audit of Continuous Source Emission Monitors (Revised Traceability Protocol No. 1)", 1987.

FIGURE 100-1
SAMPLING SYSTEM

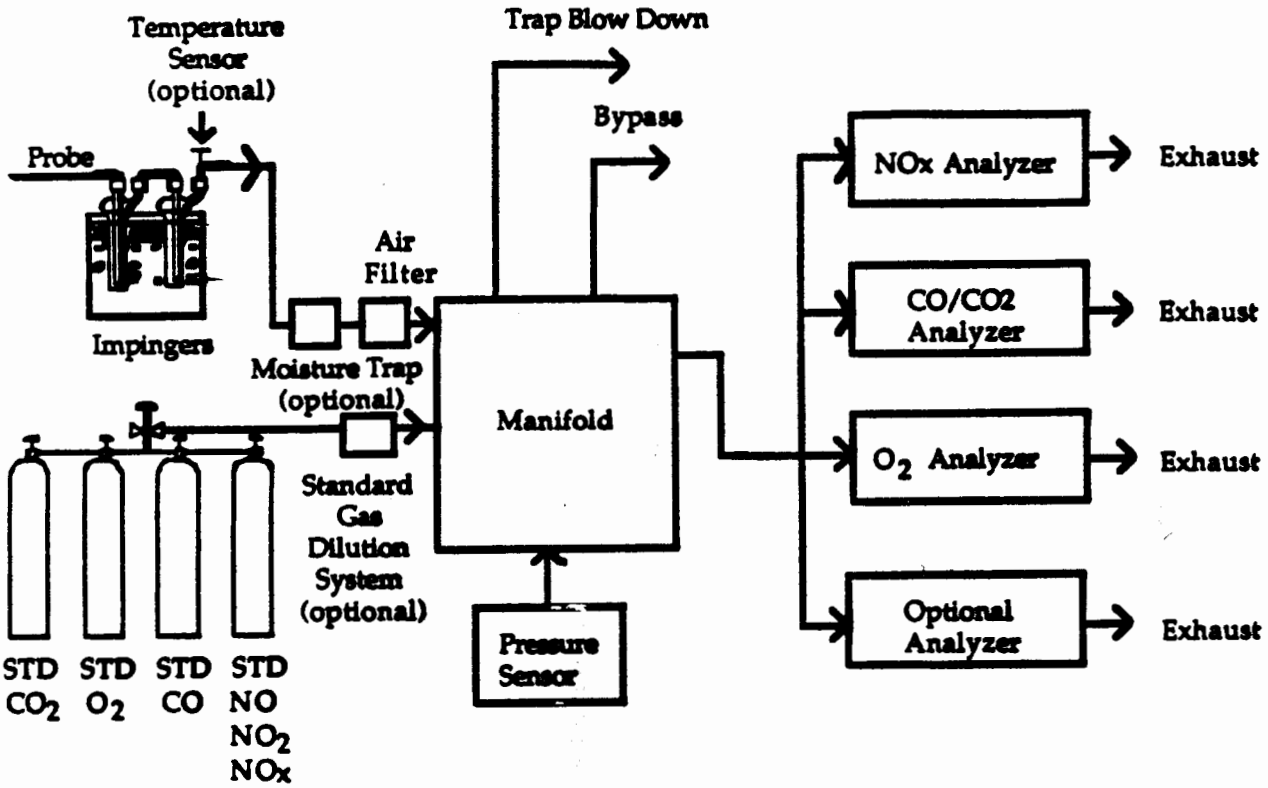

FIGURE 100-2

SITE SURVEY DATA

Date _____ P/O _____ Survey by _____

Name of company _____

Address _____

Phone number _____ Site contact _____

Entry requirements _____

Parking place _____

Location of process (distance) _____

Port access _____

Electrical circuits (3) accessible _____

Voltage _____ Amps _____

Operating schedule _____

Process description _____

Type of control unit _____

Limits: CO _____ NO_x _____ O_2 _____

Ambient conditions at the test site _____

Safety hazards _____

Required safety equipment _____

Hospital _____

Port locations/size _____

Port diagram (sketch): Show stack diameter and approximate distance to upstream and downstream flow obstructions

Additional information: Attach map to site, special instructions, etc.

FIGURE 100-3

STRIP CHART DATA
(TESTS PERFORMED BY APCD)

Date: _____ Site: _____ Test#: _____

Limits: CO _____ NO_x _____ P/O#: _____

APCD Personnel: Team Leader: _____ Others: _____

APCD Engineer: _____ Site Personnel: _____

AMB. TEMP.: _____ CHART SPEED: _____ cm/hr SAMPLE VAC _____

SPECIES	COLOR	SCALE	CYL#	CONC.	R.TIME	FLOWRATE
NO	red (for example)					
NO_2	red					
CO	green					
O_2	blue					

Name of instruments: _____ County Prop. #'s: _____

_____

_____

_____

_____

(Copy of above to be attached to chart and filled out on site)

FIGURE 100-4

STANDARD CHART NOTATIONS

	Task		Location on Chart	Symbols
1.	Chart operator's initials			(Examples below)
2.	Information from Figure 100-3		Beginning	
2.	Arrive on site:	Time _____	Left hand	0800 (use 24-hour time)
3.	Instruments turned on:	Time _____	margin	(Start): 0815
4.	Zero Recorder			Rec.
5.	Offsets (if any)			Offset
6.	Zero Instrument(s)			Zero
7.	Sample line vacuum Reading			Vac
8.	Calibrate Instrument(s)			Conc or % Dil
9.	System Bias Check			Bias

10. Comments: Note any anomalous behavior on the chart. A copy is included with/in the final report. Any changes in the engineering parameters of the equipment should be recorded if a District engineer is not monitoring these parameters.

Site: _____ PO Limits: NOx: _____
PO#: _____ ID#: _____ CO: _____
Date: _____ APCD _____
Test#: _____ SITE _____

Calibration Gases:

Gas	Cylinder	Manufacturer	Concentration	Offset (CD)	Man Press (" H20)	Man Flow (L/min)
NO						
NO						
CO						
O2						
N2						
Zero Air			Sample Vacuum:		_____ "Hg	

SGD FIELD EVALUATION (Limits: all differences +/-2%)

Gas used: ppm Scale used: ppm

Gas conc.	Inj. #1	Inj. #2	Inj. #3	Average
(CD)				
Calculated value (CD)				
% diff. of avg. from calc. val.				
% diff. from avg.				
(CD)				
Calculated value (CD)				
% diff. of avg. from calc. val.				
% diff. from avg.				
(CD)				
Calculated value (CD)				
% diff. of avg. from calc. val.				
% diff. from avg.				

MID-LEVEL GAS CONCENTRATION DIRECT TO ANALYZER (Limit: difference +/-2%)

Gas used: ppm Scale used: ppm

	Inj. #1	Inj. #2	Inj. #3	Avg.
Instrument response (CD)				
Certified value (CD)				
			Diff. %	

MULTI-POINT CALIBRATION CHECK (Limit: instrument value within 2% full scale of predicted value)

	Actual Concentration			Instrument Response (CD)		
	NO	CO	O2	NO	CO	O2
HIGH						
LOW						
MID						
			Slope			
			Intercept			
			Mid (predict.)			
			Difference (% full scale)			

PO#: TEST#:

Time _____

RUN # _____

SYSTEM CALS:

Gas	cond/ dil %		INITIAL Read (Ch Div)	INITIAL Conc. (ppm)	Syst Bias (%)	FINAL Read (Ch Div)	FINAL Conc. (ppm)	Syst Bias (%)	AVG. Conc (ppm)	ACTUAL Conc. (ppm)	Drift (%FS)
NO		direct									
		zero									
		system									
		zero									
CO		direct									
		zero									
		system									
		zero									
O2 as %		direct									
		zero									
		system									
		zero									

Sample Vac _____ "Hg

Analyzer Range: NOx 0-_____ ppm
CO 0-_____ ppm
O2 0-_____ %

EMISSION VALUES:

GAS	CH DIV	CONC. ppm uncorrected	CONC. ppm corrected*	
NOx AVG				
NOx PK1				
NOx PK2				
CO AVG				
CO PK1				
CO PK2				
O2 AVG				conc as %

*Corrected for drift and system bias.

Site: _____ PO Limits NOx: _____
PO#: _____ ID#: _____ CO: _____
Date: _____ APCD _____

Calibration Gases

Gas	Cylinder	Manufacturer	Concentration	Offset (CD)	Man. Press (" H20)	Man. Flow (L/Min)
NO						
NO						
NO2						
CO						
O2						
N2						
Zero Air						

Sample Vacuum _____ "Hg

SGD FIELD EVALUATION (Limits all differences +/-2%)

Gas used: _____ ppm Scale used: _____ ppm

Gas conc.	Inj. #1	Inj. #2	Inj. #3	Average
(CD)				
Calculated value (CD)				
% diff. of avg. from calc. val.				
% diff. from avg.				
(CD)				
Calculated value (CD)				
% diff. of avg. from calc. val.				
% diff. from avg.				
(CD)				
Calculated value (CD)				
% diff. of avg. from calc. val.				
% diff. from avg.				

MID-LEVEL GAS CONCENTRATION DIRECT TO ANALYZER (Limits differences +/-2%)

Gas used: _____ ppm Scale used: _____ ppm

	Inj. #1	Inj. #2	Inj. #3	Avg.
Instrument response (CD)				
Certified value (CD)				
			Diff. %	

NO2 CONVERTER EFFICIENCY CHECK (ON-SITE) (Limits > 90% efficiency).

	NOx MODE	NO MODE
NO2 DIR (CD)		
NO2 ZERO (CD)		
CONV EFF. %		

NO2 gas _____ ppm
Scale 0- _____ ppm

MULTI-POINT CALIBRATION CHECK (Limits Instrument value within 2% full scale of predicted value)

	Actual Concentration			Instrument Response (CD)		
	NO	CO	O2	NO	CO	O2
HIGH						
LOW						
MID						
Slope						
Intercept						
Mid (predict.)						
Difference (% full scale)						

PO#: TEST#:

Time: _____

RUN # _____

SYSTEM CALS: Gas	conc/ dil %		INITIAL Read (Ch Div)	INITIAL Conc. (ppm)	Syst Bias (%)	FINAL Read (Ch Div)	FINAL Conc. (ppm)	Syst Bias (%)	AVG. Conc (ppm)	ACTUAL Conc. (ppm)	Drift (% FS)
NO		direct									
		zero									
		system									
		zero									
NO2		direct									
		zero									
		system									
		zero									
CO		direct									
		zero									
		system									
		zero									
O2 as %		direct									
		zero									
		system									
		zero									

Analyzer Ranges: NOx 0-_____ppm Sample Vac _____ "Hg
CO 0-_____ppm
O2 0-_____%

EMISSION VALUES:

GAS	CH DIV	CONC. ppm uncorrected	CONC. ppm corrected*	
NOx AVG				
NOx PK1				
NOx PK2				
NO AVG				
NO2 AVG				
CO AVG				
CO PK1				
CO PK2				
O2 AVG				conc as %

Stack NO2 %

*Corrected for drift and system bias.

FIGURE 100-7

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

MEMORANDUM

SUBJECT: EPA Approval of San Diego APCD Test Method 100

FROM: William Grimley
Source Characterization Group B (MD-19)

TO: Duane James
Rulemaking Section, Region IX (A-5-3)

At your request, members of the Emission Measurement Center have reviewed the February 1995 draft of the subject test method entitled, "Test Procedures for the Determination of Nitrogen Oxides, Carbon Monoxide and Diluent Gases by Continuous Emission Monitoring." This method encompasses EPA Test Methods 3A, 7E, 10, and 20, in 40 CFR Part 60, Appendix A [except for SO2]. A procedure for determination of stack gas stratification was also included in the draft method. As no test data that compares the candidate method with any of the corresponding EPA methods was submitted, the method has only been reviewed from the standpoint of comparison with the technical procedures and performance specifications contained in the individual EPA methods.

The principles of sample extraction and analytical detection in candidate Method 100 are essentially the same as those in the corresponding EPA Methods. No interference response tests are specified; however it would be reasonable to exclude them as long as the method is only applied to combustion sources, and the discrimination ratios for potential interferents for the particular instruments used are known to be good.

The technical performance specifications in Method 100 are at least as rigorous as those contained in the EPA Methods, and more requirements have been added in Method 100 in respect to the determination of sample system bias and NO_2 converter efficiency. Based on these findings, we believe it would be reasonable to approve this method for application to combustion sources.

Should there be any further questions, please feel free to contact me at (919) 541-1065.