SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

PROTOCOL FOR THE MEASUREMENT OF NITROGEN OXIDES, CARBON MONOXIDE, AND OXYGEN FROM SOURCES SUBJECT TO SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT RULE 1146

SEPTEMBER 21, 2001

MONITORING & SOURCE TEST ENGINEERING BRANCH
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MONITORING & ANALYSIS
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**ATTACHMENT I**  ADDITIONAL QA/QC FOR NON-LAP APPROVED SOURCE TEST LABORATORIES

**ATTACHMENT II**  DISTRICT METHOD 100.1 GENERAL REQUIREMENTS AND NO2 TO NO CONVERTER EFFICIENCY CHECK PROCEDURE

**ATTACHMENT III**  EPA CONDITIONAL TEST METHOD CTM-030
PROTOCOL FOR THE MEASUREMENT OF NITROGEN OXIDES, CARBON MONOXIDE, AND OXYGEN FROM SOURCES SUBJECT TO SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT RULE 1146

1.0 OVERVIEW AND APPLICABILITY

On November 17, 2000, the South Coast Air Quality Management District (District) amended Rule 1146, which limits emissions of nitrogen oxides (NOx) and carbon monoxide (CO) from industrial, institutional and commercial boilers, steam generators, and process heaters with rated input capacity greater than 5 million Btu per hour. Compliance with this rule is based on District standard reference methods (Method 100.1, or Methods 7.1 and 10.1), or equivalent and approved methods that allow the use of hand-held portable monitors. This protocol has been developed to ensure standardization of test procedures including the use of: specified test conditions, required test methods, specifications for test equipment, data collection/reporting, and quality assurance requirements.

An independent testing laboratory, which meets the requirements of South Coast Air Quality Management District's Rule 304, Subdivision (k), may conduct the test. The source testing contractor must meet the non-conflict of interest requisite specified per Rule 304(k) and provide a non-conflict of interest statement in the final report. The source testing contractor need not be approved under the SCAQMD Laboratory Approval Program (LAP). If not LAP approved, detailed information including, but not limited to, the contractor's QA/QC procedures, resumes of source testers, description of the contractor's sampling system including the sampling system for SCAQMD Method 100.1, description of instruments used, and calibration procedures for all instruments are to be included in the final report for review (see Attachment I). The laboratory shall, in conducting the test, prepare a report of findings, including all raw data sheets/charts, and
analytical data. Testing must demonstrate to the satisfaction of the Executive Officer that the test data was obtained using the protocol described herein.

When equipment at a facility subject to District Rule 1146 cannot be tested using this protocol, the protocol may be modified following written approval of the Executive Officer.
2.0 ENVIRONMENTAL TEST CONDITIONS

2.1 AMBIENT TEMPERATURE
The temperature shall be monitored and recorded at fifteen minute intervals during each test.

2.2 AMBIENT RELATIVE HUMIDITY
The relative humidity shall be monitored and recorded before and after each test.

2.3 AMBIENT BAROMETRIC PRESSURE
The barometric pressure shall be monitored and recorded before and after each test.
3.0 COMBUSTION EQUIPMENT AND PROCESS DESCRIPTION

3.1 COMBUSTION EQUIPMENT DESCRIPTION
The type of combustion equipment (i.e., industrial boiler, steam generator, etc.), and control equipment shall be noted. Additionally, the rated heat input capacity and fuel type shall be recorded. Tester shall record the equipment make, model, and serial number.

Information regarding the fuel flow meter(s) which includes the make, model, serial number, and the most recent calibration date, shall also be recorded. The fuel meter calibration data shall be included with the final test report.

3.2 PROCESS DESCRIPTION
A description of the process operation during sampling shall include, but not be limited to, the following information:

a) Provide a stepwise description explaining the entire process operation (relating only to the combustion equipment). The description shall also include a schematic of this process;

b) Indicate whether the operation was a continuous or batch process. If it was a batch process, state the length of each batch interval, and when (within this interval) the test performed;

c) If the equipment modulated during sampling, indicate the extent of modulation with respect to firing rate;

d) State whether the equipment had operated normally;

e) Report the amount of product (i.e.- steam) supplied by the equipment;

f) If the equipment is a process heater, identify the product being heated;
g) Include miscellaneous data, such as steam pressure, fuel pressure and temperature, firing rate, and the percent of rated capacity at which the combustion equipment operated during testing.

h) Provide information regarding the operating schedule of the equipment. Include information such as whether the equipment operates "on demand", the peak periods of operation, and the operational hours in hours per day, days per week, and weeks per year.

i) Submit information on the pollution control equipment, including the type of control (low NOx burners, flue gas recirculation, selective catalytic reduction, etc.), control parameters (flue gas recirculation rate, ammonia injection rate, temperature of inlet gas to selective catalytic reduction equipment, etc.), and manufacturer of the control equipment.
4.0 ANALYTICAL REQUIREMENTS AND SAMPLING PROCEDURES USING STANDARD DISTRICT METHODS

4.1 APPLICABILITY

This section applies to the use of District Methods 100.1, or District Methods 7.1 and 10.1.

4.2 DISTRICT METHOD 100.1

Simultaneous and concurrent stack gas measurements of NOx, CO, and oxygen shall be determined by following District Method 100.1, and using the following types of analyzers:

<table>
<thead>
<tr>
<th>Gas Constituent</th>
<th>Analyzer Principle</th>
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<tbody>
<tr>
<td>NOx</td>
<td>Chemiluminescent</td>
</tr>
<tr>
<td>CO</td>
<td>Non-Dispersive Infrared (NDIR)</td>
</tr>
<tr>
<td>Oxygen</td>
<td>Electrochemical</td>
</tr>
</tbody>
</table>

The sample concentration must fall within 20 to 95% of the analyzer range for at least 95% of the time. To determine the best analyzer range, a preliminary screening of the gas concentration may be necessary. Since the carbon dioxide and oxygen concentrations in combustion products are related stoichiometrically, it is strongly recommended that carbon dioxide also be measured during gas sampling to partially verify the data quality. Carbon dioxide sampling is also addressed in District Method 100.1.

4.2.1 SAMPLE CONDITIONING REQUIREMENTS

For Method 100.1 testing, proper sample conditioning is essential for representative sampling. Sample conditioning includes removal of particulate matter and moisture in the
sample gas stream without removal of the pollutants of interest. In particular, NO₂, due to its high solubility in water, is more susceptible to scrubbing than, for example, NO or CO.

The District recommends a gas sampling system which can be used universally. The set up includes a heated 1/4 inch stainless steel probe with 50-80 μ (micron) size sintered 316 stainless steel or ceramic filter at the tip and a short (not more than 6 feet) heated Teflon line to a sample conditioning system. The temperature of the probe and the Teflon line should be maintained at about 250 °F. The conditioning system consists of a pair of standard Greenburg-Smith impingers with the stems cut to about 1 inch length from the top, immersed in a bath containing water and dry ice pellets, and immediately followed by a thermo-electric cooler or permeation drier. The gas temperature at the outlet of the impinger shall be less than 60 °F, and the gas at the drier outlet shall be maintained at a dew point less than 37 °F. If the drier cannot be directly connected to the impinger outlet then a Teflon line heated to 10 °F above the impinger outlet gas temperature can be used for connection. Another particulate filter (~ 5 μ) should be in line immediately after the cooler/drier. All sample conditioning temperatures should be measured and recorded preferably on the strip chart at least every 10 minutes. If the moisture content of the exhaust gas is below 5% and the sample gas flow rate is less than 10 liter/minute, the impinger set-up need not be used as long as no moisture condensation occurs in the system and the conditioned sample is maintained at the required dew point.

Precaution: Never let the water in the impingers accumulate more than 1/4 of the impinger's height. Do not let the water bath freeze around the impingers; it may cause cracking of glass impingers. Make sure that the thermo-electric cooler/permeation drier has adequate design capacity. A good maintenance schedule should be followed for the cooler/drier.
An example of a non-universally applicable water removal system is a refrigerated cooling coil based system. A refrigerated cooling coil system scrubs out a high percentage of the highly water soluble pollutants due to comparatively high residence time and intimate contact between the sample gases and water droplets collected on the inside wall of the coil. This type of sample conditioner is not suitable in all cases.

Other systems may be used, upon District’s approval, as long as they meet the requirement for water removal immediately after separation from the gas stream and are designed to minimize water contact with the gas stream.

4.2.2 INSTRUMENTAL ANALYZER PROCEDURES

A general guideline for conducting Method 100.1 is included in Attachment 11. The guideline is provided to clarify and emphasize some of the key points of Method 100.1 requirements.

Leak checks must be performed before and after each concentration sampling period. Follow the leak check procedure as stated in Method 100.1.

Analyzer calibration error, system bias and linearity checks must be performed before and after each concentration sampling period. A system response time test must be performed before the concentration sampling. Tester shall survey possible interferents emitted by the equipment, and perform interference response tests, if necessary.

NO₂ to NO converter efficiency check must be performed at the beginning of each test day. The converter efficiency check procedure is included in Attachment 11.

4.2.3 FIELD SAMPLING PROCEDURE

Field sampling shall follow the procedures in Section 2.6 of District Method 100.1. All QA/QC requirements of Method 100.1 shall be strictly followed. All field and laboratory
data, including calculations, annotated chart traces, and calibration results, shall be submitted with the test report.

Record the process variables listed in Section 3.2. For combustion equipment with a totalizing fuel meter, volumetric readings shall be recorded at the beginning and end of the test, and once every three minutes during sampling. Combustion equipment with non-totalizing fuel meters shall have readings recorded at the beginning and end of the test, and once every minute during sampling.

4.2.4 DATA ACQUISITION SYSTEM (DAS)

A DAS must be used to report the analyzer readings. The DAS must provide every one-minute and 15-minute averages in units of ppm for \( \text{NO}_x \) and CO, and in percent for \( \text{O}_2 \) (and \( \text{CO}_2 \), if desired). The strip charts must be included in the final report as well as the DAS output.

4.3 DISTRICT METHODS 7.1 AND 10.1

Simultaneous and concurrent stack gas bulb samples of NOx, CO, and oxygen shall be analyzed by the following procedures:

<table>
<thead>
<tr>
<th>Gas Constituent</th>
<th>Analytical Principle</th>
</tr>
</thead>
<tbody>
<tr>
<td>NOx</td>
<td>Colorimetric or Ion Chromatograph</td>
</tr>
<tr>
<td>CO</td>
<td>Gas Chromatograph/ Non-Dispersive Infrared (GC/NDIR)</td>
</tr>
<tr>
<td>Oxygen</td>
<td>Gas Chromatograph-Thermal Conductivity (GC/TCD)</td>
</tr>
</tbody>
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It is noted that an Orsat analysis for oxygen is not acceptable. Since the carbon dioxide and oxygen concentrations in combustion products are related stoichiometrically, it is
strongly recommended that carbon dioxide also be measured during gas sampling to partially verify the data quality. Carbon dioxide sampling is addressed in District Method 10.1.

4.3.1 FIELD SAMPLING PROCEDURES
Separate Methods 7.1 and 10.1 bulb samples shall be collected from a purged heat-traced sample line at three minute intervals, using the locations described in Section 5.4.1 of this protocol. For each bulb sample, identify the traverse point and record the sample times per Section 5.4.2. Field sampling shall follow procedures described in Section 2 of District Methods 7.1 and 10.1. Record the process variables and equipment firing rate per Section 4.2.3 of this protocol.

All QA/QC requirements of District Methods 7.1 and 10.1 shall be strictly followed. All field and laboratory data, including calculations, analytical chart traces (with annotations), and calibration results, shall be submitted with the test report.

4.3.2 LOW CONCENTRATION CO
Additional QA requirements are necessary for Method 10.1 if the CO concentration is less than 10 ppm. These requirements are:

1) The bulbs or canisters shall be purged with zero gas (nitrogen or hydrocarbon-free air) before taking any samples or blanks. Analyze the purged zero gas for CO, and reject any container that has more than 0.5 ppm CO.

2) Two field blanks of zero gas containing less than 0.5 ppm of CO in clean bulbs or canisters shall accompany the samples to the test site. These blanks shall be analyzed along with the field samples.

3) The CO shall be analyzed by Total Combustion Analysis using Flame Ionization Detection (GC/FID) according to Section 5 in District Method 25.1.
4) Analyze all samples within 24 hours of sampling.

4.3.3 LOW CONCENTRATION NOx

Additional preparation and QA requirements are necessary for Method 7.1 if the NOx concentration is less than 20 ppm. These requirements are:

1) Low NOx concentrations shall be analyzed by ion chromatography using the Alternative Laboratory Procedure described in Section 5 of District Method 7.1.

2) Sample bulbs shall be rinsed clean with 0.1 N H₂SO₄ and deionized water before sampling. Rinse, soak, and re-rinse any rubber tubing in deionized water before sampling.

3) The absorbing solution of 0.1 N H₂SO₄ with 3% H₂O₂ shall be prepared daily from freshly distilled or deionized water.

4) Establish and report the detection limit as nitrate for each set of analyses.

5) Adjust the calibration curve downwards to bracket all samples.

6) Do not store samples more than three days before analysis.

7) Analyze replicate samples and average the results.

8) Take at least one field blank sample per set to determine if contamination occurred. Prepare a bulb with absorbing solution, and fill the bulb in the laboratory with zero gas to ambient pressure. Take the bulb into the field during testing, and return the bulb to the laboratory for analysis.
5.0 ANALYTICAL REQUIREMENTS USING PORTABLE ANALYZER METHOD

Portable analyzer sampling and analysis relies on EPA Conditional Test Method CTM-030. A copy of this method is included in Attachment III.

5.1 START UP

5.1.1 ANALYZERS
Allow analyzers to warm up according to manufacturer's instructions, and Section 7.2 of the EPA document, CTM-030.

5.1.2 MOISTURE REMOVAL SYSTEM
Energize sample pump and sample line. Allow temperatures and flows to come to equilibrium. The dew point of the conditioned gas shall not be greater than 50°F. Testing shall not be performed if the ambient temperature falls below 55°F.

5.2 ANALYZER CALIBRATION
Use NIST traceable calibration gases which are certified to a minimum accuracy of ± 1%. A high span calibration gas shall be selected according to CTM-030 procedures. Calibrate the analyzers according to CTM-030 using zero gas, mid-level gas, and high-range gas. Calibrations of each analyzer must be performed prior to and following each test on the day of sampling. Linearity may be checked using a gas divider if it is calibrated according to EPA Method 205.

The analyzers shall meet the zero calibration error, span calibration error, interference response, linearity, and stability check response specifications of CTM-030, Section 4. A fresh air purge shall be performed per manufacturer's recommendations between each stability check. The pre- and post-test zero and span calibration error checks must be performed at the sampling location according to Section 7.3 and 7.6 of CTM-030.
5.3 DATA RECORDING

The data shall be recorded using procedures in Section 5.1.10 of CTM-030. The recorder must allow each data point to be read at least once every minute.

5.4 FIELD SAMPLING PROCEDURE

5.4.1 SAMPLE POINT LOCATIONS

Traverse sampling shall be performed at five points across perpendicular axes of the exhaust stack. These points are: 16.7 and 83.3 percent of the diameter along the first axis; 16.7 and 83.3 percent of the diameter along the second axis; and, the center point.

5.4.2 TRAVERSE SAMPLING

At the start of the test, record the time and fuel flow. Do not interrupt the flow to the portable analyzer. Follow the Sample Collection procedures of CTM-030 (Section 7.5) with the following exception: at each traverse location, gas concentrations shall be recorded at one minute intervals for three consecutive minutes. Note that although fifteen minutes of testing will be conducted, the actual sampling time will be longer, depending on the response time or stability time. Identify the traverse points being sampled along with the gas concentrations at each point. Determine the arithmetic mean of the gas concentrations measured during the fifteen minute test period. If required, collect a sample of fuel for composition analysis. Record the process variables and equipment firing rate per Section 4.2.3.

5.4.3 CELL TEMPERATURE MONITORING

Not required if sensors are temperature controlled. Sensor temperature shall not exceed 125 degrees F.
5.4.4 INTERFERENCE VERIFICATION AND RE-ZEROING

Refer to Sections 7.7 and 7.8 of CTM-030. In addition to the interference checks required by CTM-030, an interference check of the NO and NO2 sensors shall be performed for combustion devices which have a potential to emit SO2 concentrations greater than 10 ppm. The interference check procedure should consider both positive and negative biases in the data, and shall be submitted for District written approval prior to performing the source test. Results of the interference check shall be included with the final report, and shall demonstrate (using an equation similar to that in Section 6.3.1 of CTM-030) that the combined NO and NO2 interference responses due to SO2 contribute less than 5% of the NOx concentration in the stack.
6.0 TESTING UNDER NON-IDEAL CONDITIONS

The following is a discussion of some common non-ideal conditions and their solutions in source testing:

If there are fluctuations in the process or operating conditions, such as, changes in load, the testing may continue as long as the operating conditions are recorded to show each fluctuation. If the combustion equipment shuts down completely or if there are severe fluctuations during sampling, testing must be repeated for a minimum of 15 continuous minutes during acceptable operating conditions. The severity of fluctuations will be determined on a case-by-case basis. If the device has the tendency to operate at a non-ideal condition for testing, a protocol for testing at such condition should be submitted for review and approval prior to testing.

If there are dampers or bypass stacks present, testing should be conducted as follows:

1) If excess air is frequently introduced to the exhaust stack at a variable rate, concentration testing shall be performed while no excess air is introduced to the exhaust stack.

2) When all or part of the exhaust gas is frequently restricted to or bypassed from the exhaust stack, concentration testing shall be performed while the total exhaust gas is routed to the exhaust stack (except for flue gas recirculation).

All changes in process and operating conditions and test interruptions must be noted with the beginning and ending times of each occurrence on the field data sheets.

For multiple stacks, perform sampling at each of the stacks during acceptable testing conditions. Sampling at each stack need not be performed simultaneously.
If other non-ideal testing conditions exist, the facility must submit a source test protocol for review and approval prior to testing.
7.0  CALCULATIONS

Calculations should be carried out to at least one significant digit beyond that of the acquired data and then should be rounded off after final calculation to three significant digits for each run. All rounding off of numbers should be in accordance with the ASTM E380-82 procedures.

7.1  HEATING VALUE

If heating value of the fuel is measured continuously using an instrument, such as a calorimeter, this value shall be reported in units of Btus/scf for gaseous fuels, and Btus/gal for liquid fuels. The density (lb/gal) shall also be reported for liquid fuels.

Note that if the heating value is determined from a fuel gas composition analysis, the calculated heating value must be corrected to a standard temperature of 60 degrees Fahrenheit. The following methods may be used for fuel analysis:

- Gaseous Fuel: ASTM D1945
- Refinery Fuel: ASTM D1946
- Liquid Fuel: ASTM D2382-88

Other ASTM methods may be used upon approval by the District. Fuel analysis must be conducted by an independent laboratory.

7.2  F-FACTOR CALCULATION

For fuels not listed in Table 19-1 (EPA 40 CFR 60 Appendix A, Method 19), an F-Factor may be determined using procedures described in paragraph 3.2 of Method 19. An explanation of the analytical procedures used for determining the ultimate analysis for the fuel shall be provided with the test report.
The F-Factor shall not be used in cases where enriched oxygen is used, non-fuel sources of carbon dioxide are present (e.g., lime kilns and calciners), and the oxygen content of the stack is 19 percent or greater.

7.3 EMISSION RATE OF NO\textsubscript{x} AND CO

7.3.1 CONCENTRATION CORRECTIONS

All emission concentrations determined using District Method 100.1 or hand-held portable analyzers shall be corrected according to Section 2.7 of District Method 100.1, or per Section 8.1 of CTM-030.

For hand-held portable analyzers, NO\textsubscript{x} data less than 10 ppm shall be reported as "< 10 ppm", and then corrected per Section 8.1 of CTM-030. However, if the correction reduces the concentration value (i.e. < 9 ppm), the result shall be reported as "< 10 ppm".

7.3.2 CONCENTRATION ADJUSTMENT OF PORTABLE ANALYZER DATA

In addition to the correction provided in Section 7.3.1 above, the measured concentrations shall be adjusted as follows:

\[
\text{Adjusted Concentration} = \text{Raw Concentration} + (0.01) (T_{Err}) (\text{High Span Conc.})
\]

Where:

\[
\text{Raw Concentration} = \text{Averaged 15 minute data of the NO}_x, \text{CO, or O}_2 \text{ gas measured during stack traverse;}
\]

\[
\text{High Span Conc.} = \text{the high span calibration gas used to calibrate the NO}_x, \text{CO, or O}_2 \text{ analyzer;}
\]

\[
T_{Err} = \sqrt{E_{Cal}^2 + E_{Lin}^2 + E_{stab}^2}
\]

\[
E_{Cal} = \text{higher of the pre-test and post-test calibration errors (\%)};
\]

\[
E_{Lin} = \text{higher of the pre-test and post-test linearity errors (\%)};
\]

and,
\( E_{\text{stabl}} \) = the stability check error (\%), adjusted for a 30-minute period.

### 7.3.3 NOx OR CO EMISSION (Concentration at 3% \( \text{O}_2 \))

Compute using the following:

\[
N = (P) \cdot \frac{20.9 - 3}{20.9 - \%O_2}
\]

Where:

- \( N \) = NOx or CO Concentration (ppm) corrected to 3% \( \text{O}_2 \);
- \( P \) = NOx or CO Concentration in flue gas, (ppm), dry basis, adjusted per Sections 7.3.1 and 7.3.2, as applicable;
- \( \%O_2 \) = Oxygen concentration (\%), dry basis, measured in the flue gas and adjusted per Sections 7.3.1 and 7.3.2, as applicable.

### 7.3.4 NOx OR CO EMISSION (lb/MMBtu)

Compute using the following:

\[
E = (K) \cdot \frac{20.9}{20.9 - \%O_2} \cdot (F_d)(P)
\]

Where:

- \( E \) = NOx or CO Emission Rate (lb/MMBtu);
- \( K \) = 1.195 \times 10^{-7} \) for NOx, and 7.27 \times 10^{-8} \) for CO;
- \( \%O_2 \) = Oxygen concentration (\%), dry basis, measured in the flue gas and adjusted per Sections 7.3.1 and 7.3.2, as applicable.
- \( F_d \) = Oxygen based F-Factor, dry basis; and,
- \( P \) = NOx or CO Concentration in flue gas, (ppm), dry basis, adjusted per Sections 7.3.1 and 7.3.2, as applicable.
8.0 HIGH OXYGEN CONCENTRATIONS

Because the oxygen correction term is in the denominator of the equations presented in Section 7.3.3 and 7.3.4, errors in oxygen measurements at concentrations approaching 20.9% are greatly magnified in the calculation of NOx or CO emissions. Hence, an alternative procedure for accurately determining NOx and CO emissions at high oxygen concentrations is necessary. This section defines a procedure for determining Rule 1146 compliance (in units of lb/MMBtu) at stack oxygen concentrations exceeding 19%.

8.1 EMISSION SAMPLING

The procedures outlined in Section 4.2 shall be strictly followed. Sampling shall be conducted over a continuous period of not less than 30 minutes.

8.2 FLOW DETERMINATION

8.2.1 VELOCITY TRAVERSE

The exhaust flow rate shall be determined in order to calculate the pollutant mass emission rate. For stacks, or exhaust pipes, without existing sampling ports, determine the best sampling port location using SCAQMD Method 1.1. In order to satisfy Method 1.1, the exhaust pipe may need to be lengthened. If so, a temporary alteration may be made by attaching to the exhaust pipe an extension of the same size and shape as the original pipe, where the connecting seam does not cause a flow disturbance and is leak free.

Traverse sampling is necessary for the determination of stack gas velocity. The number of traverse points must be determined in accordance with SCAQMD Method 1.1 for stacks with diameters greater than 12 inches and SCAQMD Method 1.2 for stacks with diameters 4 to 12 inches. All traverse points, as determined by Method 1.1 or 1.2, must be used for measuring stack gas velocity.
If conditions such as negative flow and/or severe cyclonic flow exist that reduce the accuracy and precision of velocity measurements, an alternative location must be chosen where this condition does not exist.

### 8.2.2 STACK FLOW RATE

To determine the average stack flow rate in the stack, use one of the following methods:

<table>
<thead>
<tr>
<th>Method</th>
<th>Description</th>
<th>Usage</th>
<th>Limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Method 2.1</td>
<td>Determination of Stack Gas Velocity and Volumetric Flow Rate</td>
<td>Most commonly used. For stacks greater than 12 inches in diameter and velocity head greater than 0.05 in. H₂O.¹</td>
<td>Cyclonic flow will result in inaccurate velocity measurements.²</td>
</tr>
<tr>
<td>Method 2.2</td>
<td>Direct Measurement of Gas Volume Through Pipes and Small Ducts</td>
<td>For pipes or ducts where velocity traverse cannot be performed due to physical limitations, i.e., low flow and size restrictions.</td>
<td>For gas temperature range of 32 to 120 °F.</td>
</tr>
<tr>
<td>Method 2.3</td>
<td>Determination of Gas Velocity and Volumetric Flow Rate From Small Stacks or Ducts</td>
<td>For velocity measurement in stacks 4 to 12 inches in diameter using a standard Pitot tube.</td>
<td>Standard Pitot tube may pose a problem in high moisture-laden or high particulate-laden gas streams.</td>
</tr>
</tbody>
</table>

¹ If the velocity head is below 0.05 in. H₂O, the source tester may propose an alternate method for measuring low velocity, such as, using a flow measuring device manufactured especially for low velocity. Use of such instruments must be approved prior to source testing.

² If cyclonic flow is present, perform the velocity traverse as specified in the District Source Test Manual Chapter X, Section 1.2.

The velocity traverse shall be performed simultaneously with concentration testing. If pulsating flow is detected during the velocity traverses, use a dampening method as described in Chapter X Section 9 of the SCAQMD Source Test Manual.
8.2.3 **EXHAUST GAS MOLECULAR WEIGHT DETERMINATION**
Carbon dioxide (CO₂) and O₂ concentrations are needed to determine the molecular weight of the exhaust gas in order to calculate the standard volumetric flow rate. These gases shall be measured using Method 100.1. All testing procedures and QA/QC requirements must satisfy those as stated in the method. Molecular weight sampling shall be performed simultaneously with the NOₓ and CO sampling.

8.2.4 **MOISTURE DETERMINATION**
In conjunction with the velocity traverses, conduct moisture sampling using Method 4.1. A single sampling point near the stack center is acceptable for moisture sampling. If low moisture is suspected (< 5%), increase the sampling time to acquire at least twice the required sample volume.

8.3 **HEAT INPUT**
8.3.1 **HEATING VALUE**
The higher heating value of the fuel shall be determined according to the procedures in Section 7.1 of this protocol.

8.3.2 **FUEL USAGE**
The fuel usage shall be determined using a totalizing fuel meter by calculating the difference between the facility's fuel meter volume readings (in cubic feet) prior to, and following the Method 100.1 sampling period. This fuel usage must be corrected to standard pressure (14.7 psia) and temperature (60°F). The time between fuel meter readings (in minutes) must also be accurately recorded.

Meter calibration data must be submitted with the final report.
8.4 CALCULATIONS

8.4.1 POLLUTANT MASS EMISSIONS

The NOx and CO mass emissions rate (in units of lb/hr) shall be determined according to the following equation:

\[ E_R = (1.583 \times 10^{-7}) (MW) (Flow) (P) \]

Where:

- \( E_R \) = NOx or CO Emission Rate (lb/hr);
- \( MW \) = Molecular weight, 46 for NOx, and 28 for CO;
- \( Flow \) = Exhaust stack flow rate (dscfm), determined using calculation procedures in District Method 2.1;
- \( P \) = NOx or CO Concentration in flue gas, (ppm), dry basis, corrected according to Section 2.7 of District Method 100.1.

8.4.2 HEAT INPUT CALCULATION

The heat input shall be calculated from data collected in Section 8.3 as follows:

\[ HI = (U)(HHV)(60)(10^6) \]

Where:

- \( HI \) = Heat input rate (MMBtu/hr);
- \( U \) = Fuel input rate (scf/min), calculated by dividing the fuel usage (Section 8.3.2) by the minutes between fuel meter readings;
- \( HHV \) = Higher heating value of the fuel, determined in Section 8.3.1.

8.4.3 NOx OR CO EMISSION CALCULATION

The NOx or CO emissions, for Rule 1146 compliance determination is calculated by:

\[ C_{R1146} = (E_R) / (HI) \]

Where:
\[ C_{R1146} = \text{NOx or CO emissions (lb/MBtu) for Rule 1146 compliance determination;} \]

\[ E_R = \text{NOx or CO Emission Rate (lb/hr), determined from Section 8.4.1;} \]

\[ H_I = \text{Heat input rate (MMBtu/hr), determined from Section 8.4.2.} \]
9.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

The test and analytical equipment shall meet all QA/QC requirements cited throughout this Protocol, and where not cited, shall be calibrated as a minimum within the requirements set forth in SCAQMD Source Test Methods Chapter III, Calibrations. The facility's equipment fuel meter shall be calibrated in accordance with procedures outlined in EPA 40 CFR Part 75, Appendix E.

9.1 PRESSURE MEASUREMENT

Pressure measurement instruments shall have an error no greater than the following values:

<table>
<thead>
<tr>
<th>Measurement</th>
<th>Accuracy</th>
<th>Precision</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas and Liquid</td>
<td>± 5% (of Reading)</td>
<td>± 2% (of Range)</td>
</tr>
<tr>
<td>Pressure</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

9.2 TEMPERATURE MEASUREMENTS

Temperature measuring instruments shall have an error no greater than the following values:

<table>
<thead>
<tr>
<th>Accuracy</th>
<th>Precision</th>
</tr>
</thead>
<tbody>
<tr>
<td>± 1.5% of Reading</td>
<td>± 1.5% of Reading</td>
</tr>
</tbody>
</table>

9.3 FUEL GAS FLOW

The gas meter reading shall be corrected to dry standard conditions (60°F, 14.7 psia), and adjusted for instrument calibration. Calibration curves shall be used if required. The overall accuracy of gas flow measurements shall be equal to or less than 2% of the actual value.
9.4 NON-GASEOUS FUEL USAGE MEASUREMENTS

The accuracy of non-gaseous fuel use measurements, using calibration curves if required, shall be equal to or less than \( \pm 2\% \) of the actual value. Calibration data shall be submitted with the test report.

9.5 TIME

The elapsed time measurement shall be measured with an instrument that is accurate within \( \pm 0.5 \) seconds per hour.
10.0 REPORT

10.1 REPORT INFORMATION

The following information is to be included in the test report:

1. All printouts, must be included in the final report and must be clearly identified as to:
   - location/source
   - operator initials
   - date/running times
   - actual test interval
   - contaminant/diluent
   - range of changes
   - range of measurement
   - calibrations
   - cal gas concentration/cyl. no.
   - range of calibration

2. A summary of the Source Test results.

3. A brief process description. Indicate equipment operation during testing, as well as any other information which may influence the final report.

4. A simple schematic diagram of the process, showing the sampling location, with respect to the upstream and downstream flow disturbances.

5. The sampling and analytical procedures. Be specific about all aspects of sampling and analysis. Include diagrams of test equipment and methods.

6. Complete raw field data, including production data indicative of the testing interval, analyses, and the test results (show all calculations).

10.2 REPORT FORMAT

The information in the final report shall be formatted as follows:

I. Table of Contents

II. Executive Summary

III. Introduction

IV. Equipment Description Including Fuel Meter(s), if applicable. This section shall include a statement that verifies acceptability of the method test location and the operating condition during the test.

V. Test Critique

VI. List of Sampling and Analytical Methods Used. This section shall include a list of the test methods used. Do not include copies or descriptions of the source test methods if the methods were adhered to as written. If exceptions were made to the methods, submit only an explanation of the exceptions.

VII. Appendices

A. SCAQMD Method Results

B. Portable Analyzer Results

C. Schematics of Stack Sampling Locations

D. Field Data Sheets - testing method and process data sheets shall be compiled in separate sections

E. QA/QC

F. Laboratory Analytical Data

G. Calibration Data and Calculations

H. Calibration Gas Certificates

I. Chain of Custody Information (as necessary)

J. Process Operating Data Including Fuel Usage During Test

K. Calculations Using Fuel F-Factor (if necessary)

L. Additional QA/QC for Non-LAP Approved Source Test Laboratories (if necessary)
ATTACHMENT I

ADDITIONAL QA/QC FOR
NON-LAP APPROVED
SOURCE TEST LABORATORIES

GENERAL INFORMATION

CERTIFICATE OF NON CONFLICT OF INTEREST
SOURCE TEST LABORATORIES
GENERAL INFORMATION

1) Name of Laboratory: ____________________________

2) Street Address: ________________________________

3) P.O. Box: ________________________________

4) City: _______________________ State: __________

5) Zip: ______________ County: ___________________

6) Phone No.: ______________ Fax No.: ______________

7) e-mail Address: ________________________________

8) Individual Owner's Name (if applicable): ______________

9) Name of Parent Company (if applicable): ______________

10) Authorized Representative responsible for ensuring that the laboratory complies with
the conditions and criteria for testing approval: ______________
    Title: ________________________________

11) Technical Director or Manager of Laboratory: ______________
    Title: ________________________________

12) List of methods for which testing was conducted:
    ________________________________
    ________________________________
    ________________________________

13) Organization
    Please attach a personnel organization chart. Please show all management staff and
    personnel who will be performing work under LAP, including technicians, student
    workers, and temporaries, and describe their duties. Be sure to identify the following
    functions however named:
Authorized contact. This person has the authority to initiate or terminate the Approval, or to change its scope. This person has the authority to commit to meeting LAP requirements.

Technical director. This person has overall technical responsibility for the test facility.

QA Officer. This person guarantees that the data quality meets test plan or project specifications, has overall responsibility to review implementation of QA policies, takes corrective action, and is not involved in production of data. This person keeps complaint records for one year.

Supervisor(s), project manager(s), or team leader(s): These people have day to day oversight of work and data quality.

14) Personnel
Please provide a curriculum vita or resume for all management staff and personnel who perform LAP work, including technicians, student workers, temporaries etc. Specifically address education, relevant experience, special training, title and normal duties.

15) Integrity
Please attach a copy of your policies on individual conflict of interest and sign the conflict of interest form included with this Application.

Please attach a copy of your policies on data falsification and data selection/rejection, and a copy of your policies on QC.

16) Chain of Custody, Evidence Retention
Please attach your Chain of Custody policy and provide a copy of the Chain of Custody form. Please describe your procedures for identifying, receiving, and storing samples, and for assuring the security of samples, data, equipment etc. Please describe your procedures for authorizing sample and data disposal.

17) Equipment, Instruments, Reagents and Standards
Please attach a copy of your procedure for determining equipment, instrument, reagent, and standard suitability and for maintaining traceability.
18) **Documentation**
Please attach a copy of your procedure for generating and validating data forms, formats, procedures, logbooks, SOPs, spreadsheets, data, reports, etc. and for maintaining revision control.

19) **Quality Assurance**
Please describe how deviations or problems in facilities, data integrity, chain of custody, security, methods, equipment, instruments, reagents, standards, quality control, documents, reports, etc are detected, flagged and corrected. Describe the role of each staff level for maintaining data quality, and describe the duties and authority of the Quality Assurance officer in detail.

20) **Please include with this application:**
20a. General description of the laboratory facility, physical location, and scope of operation. This should include size, remote sites or sub-facilities, mobile units and major equipment. This should also include an indication of the amount of testing that is performed in fields similar to the one performed in this test report. Provide a brief overview of other testing services offered by the laboratory.

20b. Year laboratory established.

20c. List of major equipment and instrument.

20d. Standard Operating Procedures (SOPs) for each method used (except portable analyzer methods). These SOPs should include step-by-step instructions for performance of the particular method.

20e. Conflict of interest statement (example supplied herewith).

20f. Signed agreement to participate in site/ proficiency audit for Rule 1146 testing.

________________________________________  __________________________
Authorized Signature                       Date
CONFLICT OF INTEREST STATEMENT

1. The test facility shall have no financial interest in the company or facility being tested, or in the parent company or any subsidiary thereof.

2. The company or facility being tested, or parent company or subsidiary thereof, shall have no financial interest in the test facility.

3. Any company or facility responsible for the emission of significant quantities of pollutants to the atmosphere, or parent company or subsidiary thereof, shall have financial interest in the test facility.

4. The test facility shall not be in partnership with, own or be owned by, in any part or in full, the contractor who has provided or installed equipment (basic or control), or monitoring systems, for the company being tested.

I certify that the test facility meets the above criteria to the best of my knowledge and belief.

________________________________________  __________
Signature, authorized contact                      Date
ATTACHMENT II

DISTRICT METHOD 100.1 GENERAL REQUIREMENTS AND NO₂ TO NO CONVERTER EFFICIENCY CHECK PROCEDURE
The District requires continuous gas monitoring equipment employing sample extraction and conditioning, and electronic detection, to be conducted strictly according to District Method 100.1, with the emphasis upon representativeness, documentation, and quality assurance. This includes, in part:

1. Gas analyzers must meet minimum acceptable standards for method of detection, sensitivity, noise, precision, linearity, and interference (see TABLE 100.1-1 for details). Also, the gas sample extraction and conditioning equipment (probe, filter, pump, conditioner, connective plumbing, etc., and data acquisition and logging equipment shall meet minimum acceptable specifications, as described in Method 100.1.

2. It is recommended that the entire sampling system for continuous gas monitoring instruments should be leak checked before and after each test run by evacuating the system to a minimum of 20 in. Hg vacuum, and plugging for a period of 5 minutes. The resultant loss of vacuum can not exceed 1 in. Hg during this period.

3. Calibration of all analyzers must be accomplished at zero, mid span (40-60% of full scale range), and high span (80-95% of full scale range). The lowest practicable range should be selected for monitoring, so that the measured emission values are within 20-95% of the range. If a significant amount of the data are outside of this range, the data may be rejected, depending upon the application.

4. The calibration gases must be certified according to EPA Protocol Number 1, or certified to an analytical accuracy of ±1% and be NIST traceable (except cal gases used for system bias check), following EPA-600/R93/224, “EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards”, TABLE 2-2. Superblend or multi-component blend gas recertifications are based upon the individual component(s) with the shortest recertification date.

5. Field calibrations employing gas dilution systems (mass flow or critical orifice) must be performed in accordance with EPA Method 205, "Gas Dilution Verification Protocol", or available “Draft” SCAQMD Method, and supported with appropriate documentation. A 5-point check is required.

6. A calibration error check, and zero/span drift check must be performed before and after each test run. Calibration error must be less than ±2% of the range of measurement for zero, mid, and high range calibration gases. Zero/span drift must be less than ±3% of the range of measurement.

7. A system bias check must be performed before and after each test run by alternately introducing cal gases to the entire sampling system, then to the gas analyzer(s), for comparison. The difference can not exceed ±5% of the analyzer range.

8. Semi-annual analyzer certifications consisting of linearity plot, calibration curve, response time, and interference response must be furnished with the other calibrations to satisfy Q/A documentational requirements.

9. NOx measurement must be performed in the NOx mode of the analyzer. An NO2 to NO converter is required if NO2 constitutes 5% or more of the total NOx in the sample stream, or
the rule or permit condition requires "NOx" monitoring. The NO\textsubscript{2} to NO converter must be at least 90% efficient (use the NO\textsubscript{2} to NO converter efficiency procedure). The converter should be high temperature (650°C) stainless steel, if no NH\textsubscript{3} is present. If NH\textsubscript{3} is present in the sample stream, then a low temperature (350°C) molybdenum catalyst must be used in the converter. This check must be done at the beginning of the test.

10. The connective tubing from the probe to the sample conditioner must be heated above the dewpoint and the dewpoint reported. The sample conditioner must be able to maintain a dewpoint temperature of 37°F or less.

11. Data recorder resolution must be at least 0.5% of the range of measurement. A data point for each contaminant/diluent monitored must be recorded at least once/minute. Analog chart recorders must have a minimum 10-inch chart width, with 100 minor divisions.

12. All facets of testing must be continuously recorded. This includes the 3-point calibration, system bias, calibration error, and zero/span drift checks, which must precede and conclude each test run.

13. All chart traces, or digital printouts, must be included in the final report and must be clearly identified as to:
   - location/source
   - operator initials
   - date/running times
   - actual test interval
   - contaminant/diluent
   - range changes
   - range of measurement
   - calibrations
   - cal gas concentration/cyl. no.
   - range of calibration

14. When more than one gas trace is shown on a chart, the individual traces must be distinguishable by color coding or some other means (original charts may be submitted, and returned following evaluation). If a gas measurement range has been "offset" from zero, or zero has been "transposed" to the right side of the recorder chart, it must be clearly identified. This offset should not be more than 5-small divisions of the chart. This data must be corrected using the ratio of the offset.

15. Gaseous measurements must be conducted a minimum of 15 continuous minutes at each load or specified condition, after the readings have stabilized (RECLAIM reference method sampling requires a minimum of 30 continuous minutes at each load for RATAs).

16. Sampling locations not meeting the minimum site selection standards for District Method 1 must be tested for absence of stratification. (A gaseous constituent concentration profile differing more than 10% between any two monitoring points within the same cross-sectional plane of a stack or duct indicates stratification.) If stratification is present, and alternate approved site selection or modification is not possible, then special monitoring (see Chapter X of the District Source Testing Manual) will be required.
NO₂ TO NO CONVERSION TEST PROCEDURE
(Alternative to O₃ Titration Method-40 CFR 50.1, Appendix F)

1. NOₓ Analyzer Requirements
   a. Full span range 0-20 ppm or 0-25 ppm
   b. Equipped with NO and NOₓ modes

2. Auditing Gas Requirements
   a. NO₂ in air (or N₂): Use NO₂ in air for a stainless steel converter.
   b. Concentration of NO₂: 15 to 18 ppm ± 0.5 ppm
   c. Recertification: An audit gas should be recertified after six months.

3. Calibration Gas Requirements
   a. Concentration: NO (17 to 19 ppm) with less than 0.1 ppm NO₂ - High Span
      NO (10 to 13 ppm) with less than 0.1 ppm NO₂ - Mid Span
   b. Zero Gas: High purity N₂

4. Calibration of Analyzer:
   a. Calibrate NO mode with the NO calibration gases.
   b. Calibrate NOₓ mode with the same gases without any gain adjustment.
   b1. If the analyzer is equipped with two independent gain adjusting circuits, skip 4.b., then repeat 4.a. for the NOₓ mode.

5. Conversion Efficiency (CE) Test
   a. Analyze the audit gas with NO mode. Read and standardize concentration. \( (C₁, \text{ ppm}) \)
   b. Analyze the audit gas with NOₓ mode. Read and standardize concentration. \( (C₂, \text{ ppm}) \)

6. Calculation for Conversion Efficiency:
\[
\%CE = \frac{|C₂ - C₁|}{C₀} \times 100
\]

7. Criteria for Acceptability of CE
   a. %CE must be larger than 90%.
   b. \( C₁ \) must be less than 5% of total NOx (NO + NO₂) in the NO₂ audit gas (Section 2b).

---

NOTE: NO₂ audit gas concentration of higher value than what is specified in Section 2 may be required where NO₂ present in the exhaust gas being measured is greater than 30 ppm. Select the NO₂ gas within 10% of the expected NO₂ concentration in the exhaust.
ATTACHMENT III

EPA CONDITIONAL TEST METHOD
CTM-030
Determination of Nitrogen Oxides, Carbon Monoxide, and Oxygen Emissions from Natural Gas-Fired Engines, Boilers and Process Heaters Using Portable Analyzers

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability. This method is applicable to the determination of nitrogen oxides (NO and NO₂), carbon monoxide (CO), and oxygen (O₂) concentrations in controlled and uncontrolled emissions from natural gas-fired reciprocating engines, combustion turbines, boilers, and process heaters. Due to the inherent cross sensitivities of the electrochemical cells, this method should not be applied to other pollutants or emission sources without a complete investigation of possible analytical interferences and a comparative evaluation with other EPA test methods.

1.2 Principle. A gas sample is continuously extracted from a stack and conveyed to a portable analyzer for determination of NO, NO₂, CO, and O₂ gas concentrations using electrochemical cells. Analyzer design specifications, performance specifications, and test procedures are provided to ensure reliable data. Additions to or modifications of vendor-supplied analyzers (e.g. heated sample line, flow meters, etc.) may be required to meet the design specifications of this test method.

2. RANGE AND SENSITIVITY

2.1 Analytical Range. The analytical range for each gas component is determined by the electrochemical cell design. A portion of the analytical range is selected by choosing a span gas concentration near the flue gas concentrations.

2.1.1 CO and NO Span Gases. Choose a span gas concentration such that the average stack gas reading for each test run is greater than 25 percent of the span gas concentration. Alternatively, choose the span gas such that it is not greater than twice the concentration.
equivalent to the emission standard. If concentration results exceed 125 percent of the span gas at any time during the sampling run then the test run for that channel is invalid.

2.1.2 \( \text{NO}_2 \) Span Gas. Choose a span gas concentration such that the average stack gas reading for each test run is greater than 25 percent of the span gas concentration. Alternatively, choose the span gas concentration such that it is not greater than the ppm concentration value of the NO span gas. The tester should be aware that \( \text{NO}_2 \) cells are generally designed to measure much lower concentrations than NO cells and the span gas should be chosen accordingly. If concentration results exceed 125 percent of the span gas at any time during the sampling run then the test run for that channel is invalid.

2.1.3 \( \text{O}_2 \) Span Gas. The difference between the span gas concentration and the average stack gas reading for each run shall be less than 10% \( \text{O}_2 \). Where the stack oxygen is high, dry ambient air (20.9% \( \text{O}_2 \)) may be used.

2.2 Sensitivity. The minimum detectable limit depends on the nominal range of the electrochemical cell, calibration drift, 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 nominal range.

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, sample transport, sample conditioning, or protection of the electrochemical cells from particulate matter and condensed moisture.
3.1.2 **External Interference Gas Scrubber.** A tube filled with scrubbing agent used to remove interfering compounds upstream of some electrochemical cells.

3.1.3 **Electrochemical Cell.** That portion of the system that senses the gas to be measured and generates an output proportional to its concentration. Any cell that uses diffusion-limited oxidation and reduction reactions to produce an electrical potential between a sensing electrode and a counter electrode.

3.1.4 **Data Recorder.** A strip chart recorder, computer, or digital recorder for recording measurement data.

3.2 **Nominal Range.** The range of concentrations over which each cell is operated (25% to 125% of span gas value). Several nominal ranges may be used for any given cell as long as the linearity and stability check results remain within specification.

3.3 **Span Gas.** A known concentration of a gas in an appropriate diluent gas.

3.4 **Zero Calibration Error.** The gas concentration exhibited by the gas analyzer in response to zero-level calibration gas.

3.5 **Span Calibration Error.** The difference between the gas concentration exhibited by the gas analyzer and the known concentration of the span gas.

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

3.7 **Interference Check.** A method of quantifying analytical interferences from components in the stack gas other than the analyte.
3.8 **Linearity Check.** A method of demonstrating the ability of a gas analyzer to respond consistently over a range of gas concentrations.

3.9 **Stability Check.** A method of demonstrating that an electrochemical cell operated over a given nominal range provides a stable response and is not significantly affected by prolonged exposure to the analyte.

3.10 **Stability Time.** As determined during the Stability check; the elapsed time from the start of the gas injection to the start of the 30-minute stability check period.

3.11 **Initial NO Cell Temperature.** The temperature of the NO cell that is recorded during the most recent pretest calibration error check. Since the NO cell can experience significant zero drift with temperature changes in some situations, the temperature must be monitored if the analyzer does not display negative concentration results.

4. **MEASUREMENT SYSTEM PERFORMANCE SPECIFICATIONS**

4.1 **Zero Calibration Error.** Less than or equal to ±3 percent of the span gas value for NO, NO₂, and CO channels and less than or equal to ±0.3% O₂ for the O₂ channel.

4.2 **Span Calibration Error.** Less than ±5% of the span gas value for NO, NO₂, and CO channels and less than or equal to ±0.5% O₂ for the O₂ channel.

4.3 **Interference Response.** The CO and NO interference responses must be less than or equal to ±5 percent of the average stack concentration for each test run.

4.4 **Linearity.** For the zero, mid-level, and span gases; the absolute value of the difference between the gas value and the analyzer response shall not be greater than 2.5% of the span gas
concentration for NO, CO and O₂ cells and not greater than 3.0% of the span gas for NO₂ cells.

4.5 Stability Check Response. The analyzer responses to CO, NO, and NO₂ span gases shall not vary more than 2.0% of span gas value over a 30-minute period or more than 1.0% of the span gas value over a 15-minute period.
5. APPARATUS AND REAGENTS

5.1 Measurement System. Use any measurement system that meets the performance and design specifications in Sections 4 and 5 of this method. The sampling system shall maintain the gas sample at a temperature above the dew point up to the moisture removal system. The sample conditioning system shall be designed so that there are no entrained water droplets in the gas sample when it contacts the electrochemical cells. A schematic of an acceptable measurement system is shown in Figure 1. The essential components of the measurement system are described below:

5.1.1 Sample Probe. Glass, stainless steel, or other nonreactive material, of sufficient length to traverse the sample points. If necessary to prevent condensation, the sampling probe shall be heated.

5.1.2 Heated Sample Line. Heated (sufficient to prevent condensation) nonreactive tubing, to transport the sample gas to the moisture removal system.

5.1.3 Sample Transport Lines. Nonreactive tubing to transport the sample from the moisture removal system to the sample pump, sample flow rate control, and electrochemical cells.

5.1.4 Calibration Assembly. A tee fitting to attach to the probe tip for introducing calibration gases at ambient pressure during the calibration error checks. The vented end of the tee should have a flow indicator to ensure sufficient calibration gas flow. Alternatively use any other method that introduces calibration gases at the probe at atmospheric pressure.

5.1.5 Moisture Removal System. A chilled condenser or similar device (e.g., permeation dryer), to remove condensate continuously from the sample gas while maintaining minimal contact between the condensate and the sample gas.
5.1.6 Particulate Filter. Filters at the probe or the inlet or outlet of the moisture removal system and inlet of the analyzer may be used to prevent accumulation of particulate material in the measurement system and extend the useful life of the components. All filters shall be fabricated of materials that are nonreactive to the gas being sampled.

5.1.7 Sample Pump. A leak-free pump, to pull the sample gas through the system at a flow rate sufficient to minimize the response time of the measurement system. The pump may be constructed of any material that is nonreactive to the gas being sampled.

5.1.8 Sample Flow Rate Control. A sample flow rate control valve and rotameter, or equivalent, to maintain a constant sampling rate within 10 percent during sampling and calibration error checks. The components shall be fabricated of materials that are nonreactive to the gas being sampled.

5.1.9 Gas Analyzer. A device containing electrochemical cells to determine the NO, NO₂, CO, and O₂ concentrations in the sample gas stream and, if necessary, to correct for interference effects. The analyzer shall meet the applicable performance specifications of Section 4. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (e.g., precision rotameter, pressure gauge downstream of all flow controls, etc.) shall be provided at the analyzer. (Note: Housing the analyzer in a clean, thermally-stable, vibration-free environment will minimize drift in the analyzer calibration, but this is not a requirement of the method.)

5.1.10 Data Recorder. A strip chart recorder, computer, or digital recorder, for recording measurement data. The data recorder resolution (i.e., readability) shall be at least 1 ppm for CO, NO, and NO₂; 0.1% O₂ for O₂; and one degree (C or F) for temperature. Alternatively, a digital or analog meter having the same resolution
may be used to obtain the analyzer responses and the readings may be recorded manually.

5.1.11 External Interference Gas Scrubber. Used by some analyzers to remove interfering compounds upstream of a CO electrochemical cell. The scrubbing agent should be visible and should have a means of determining when the agent is exhausted (i.e. color indication).

5.1.12 NO Cell Temperature Indicator. A thermocouple, thermistor, or other device must be used to monitor the temperature of the NO electrochemical cell. The temperature may be monitored at the surface or within the cell.

5.2 Calibration Gases. The calibration gases for the gas analyzer shall be CO in nitrogen or CO in nitrogen and O₂, NO in nitrogen, NO₂ in air or nitrogen, and O₂ in nitrogen.

5.3.1 Span Cases. Used for calibration error, linearity, and interference checks of each nominal range of each cell. Select concentrations according to procedures in Section 2.1.

5.3.2 Mid-Level Cases. Select concentrations that are 40-60% of the span gas concentrations.

5.3.3 Zero Gas. Concentration of less than 0.25 percent of the span gas for each component. Ambient air may be used in a well ventilated area.

6. MEASUREMENT SYSTEM PERFORMANCE CHECK PROCEDURES

Perform the following procedures before measurement of emissions (Section 7).

6.1 Calibration Gas Concentration Verification. For the mid-level and span cylinder gases, use calibration gases certified according to EPA Protocol G1 or G2 procedures (see Bibliography). Alternative
certification techniques may be used if they are approved in writing by the applicable regulatory agency.

6.2 Linearity Check. Conduct the following procedure once for each nominal range that is to be used on each electrochemical cell (NO, NO₂, CO, and O₂) before each field test program. If a field test program lasts longer than five days, the linearity check shall be repeated before each five days of analyzer operation. Repeat the linearity check if a cell is replaced.

6.2.1 Linearity Check Gases. For each cell obtain the following gases: zero (0-0.25% of nominal range), mid-level (40-60% of span gas concentration), and span gas (selected according to Section 2.1).

6.2.2 Procedure. If the analyzer uses an external interference gas scrubber with a color indicator, using the analyzer manufacturer's recommended procedure, verify that the scrubbing agent is not depleted. After calibrating the analyzer with zero and span gases, inject the zero, mid-level, and span gases that are appropriate for each nominal range to be used on each cell. Gases need not be injected through the entire sample handling system. Purge the analyzer briefly with ambient air between gas injections. For each gas injection, verify that the flow rate is constant and that the analyzer responses have stabilized before recording all of the responses on a form similar to Figure 2.

6.3 Interference Check. Following each linearity check, use the results from the span gas injections to determine interference responses for the CO and NO cells.

6.3.1 CO Interference Response.

\[ I_{CO} = \left( \frac{R_{CO-NO}}{C_{NO2g}} \times \frac{C_{NO2S}}{C_{CO2}} \right) + \left( \frac{R_{CO-NO2}}{C_{NO2S}} \times \frac{C_{NO2S}}{C_{CO2}} \right) \times 100 \]

where: \( I_{CO} \) = CO interference response (percent)
\( R_{CO-NO} \) = CO response to NO span gas (ppm CO)
\[ C_{\text{NOG}} = \text{concentration of NO span gas (ppm NO)} \]
\[ C_{\text{NOG}} = \text{concentration of NO in stack gas (ppm NO)} \]
\[ C_{\text{COG}} = \text{concentration of CO in stack gas (ppm CO)} \]
\[ R_{\text{CO-NO}} = \text{CO response to NO span gas (ppm CO)} \]
\[ C_{\text{NO2G}} = \text{concentration of NO}_2 \text{ span gas (ppm NO}_2) \]
\[ C_{\text{NO2S}} = \text{concentration of NO}_2 \text{ in stack gas (ppm NO}_2) \]
6.3.2 NO Interference Response.

\[ I_{NO} = \left( \frac{R_{NO-NO_2}}{C_{NO_2G}} \right) \times \left( \frac{C_{NO_2S}}{C_{NOxS}} \right) \times 100 \]

where:
- \( I_{NO} \) = NO interference response (percent)
- \( R_{NO-NO_2} \) = NO response to NO\(_2\) span gas (ppm NO)
- \( C_{NO_2G} \) = concentration of NO\(_2\) span gas (ppm NO\(_2\))
- \( C_{NO_2S} \) = concentration of NO\(_2\) in stack gas (ppm NO\(_2\))
- \( C_{NOxS} \) = concentration of NO\(_x\) in stack gas (ppm NO\(_x\))

6.4 Stability Check. Conduct the following procedure once for each nominal range that is to be used on each pollutant electrochemical cell (NO, NO\(_2\), and CO) before each field test program. If a field test program lasts longer than five days, the stability check shall be repeated before each five days of analyzer operation. Repeat the stability check if a cell is replaced or if a cell is exposed to gas concentrations greater than 125 percent of the highest span gas concentration.

6.4.1 Procedure. Inject the span gas into the analyzer and record the analyzer response at least once per minute until the conclusion of the test. One-minute average values may be used instead of instantaneous readings. After the analyzer response has stabilized, continue to flow the span gas for at least 30 minutes. Make no adjustments to the analyzer during the test except to maintain constant flow. Record the stability time as the number of minutes elapsed between the start of the gas injection and the start of the 30-minute stability check period. If the concentration reaches a peak value within five minutes, you may choose to record the data for at least 15 minutes following the peak.

6.4.2 Calculations. Determine the highest and lowest concentrations recorded during the 30-minute period and record the results on a form similar to Figure 3. The absolute value of the difference between the maximum and minimum values recorded during the 30-minute period must be less than 2.0% of the span gas concentration. Alternatively, record stability check data in the same manner for the 15-minute
period following the peak concentration. The difference between the maximum and minimum values for the 15-minute check must be less than 1.0% of the span gas concentration.

7. EMISSION TEST PROCEDURE

7.1 Selection of Sampling Site and Sampling Points.

7.1.1 Reciprocating Engines. Select a sampling site located at least five stack diameters downstream of any turbocharger exhaust, crossover junction, or recirculation take-offs and upstream of any dilution air inlet. Locate the sampling site no closer than one meter or three stack diameters (whichever is less) upstream of the gas discharge to the atmosphere. Use a minimum of three sampling points located at positions of 16.7, 50, and 83.3 percent of the stack diameter. Alternatively, the tester may choose an alternative sampling location and/or sample from a single point in the center of the duct if previous test data demonstrate that the stack gas concentration does not vary significantly across the duct diameter.

7.1.2 Combustion Turbines. Select a sampling site and sample points according to the procedures in 40 CFR, Part 60, Appendix A, Method 20. Alternatively, the tester may choose an alternative sampling location and/or sample from a single point in the center of the duct if previous test data demonstrate that the stack gas concentrations of CO, NOx, and O2 does not vary significantly across the duct diameter.

7.2 Warm Up Period. Assemble the sampling system and allow the analyzer and sample interface to warm up and adjust to ambient temperature at the location where the stack measurements will take place.

7.3 Pretest Calibration Error Check. Conduct the calibration error check at the sampling location (near the sampling port) just prior to the start of an emissions test or test run. Keep the analyzer in the
same location until the post test calibration error check is conducted.

7.3.1 For analyzers that use an external interference gas scrubber tube, inspect the condition of the scrubbing agent and ensure that it will not be exhausted during sampling.

7.3.2 Inject the zero and span calibration gases at the probe tip using the calibration assembly. Ensure that the calibration gases flow through all parts of the sample interface (including any exhaust lines). During this check, make no adjustments to the system except those necessary to achieve the correct calibration gas flow rate at the analyzer. Set the analyzer flow rate to the value recommended by the analyzer manufacturer. Allow each reading to stabilize before recording the result on a form similar to Figure 4. The time allowed for the span gas to stabilize shall be no less than the stability time noted during the stability check. After achieving a stable response, disconnect the gas and briefly purge with ambient air.

7.3.3 Determine the NO and CO response times by observing the time required to respond to 95% of a step change in the analyzer response for both the zero and span gases. Note the longer of the two times as the response time. For NO, span gas record the time required to respond to 90% of a step change.

7.3.4 Calibrate all electrochemical cells in the analyzer if the analyzer uses an internal calculation method to compensate for interferences.

7.3.5 If the zero and span calibration error test results are not within the specifications in Section 4, take corrective action and repeat the calibration error check until acceptable performance is achieved.

7.4 **NO Cell Temperature Monitoring.** Record the initial NO cell temperature during the pretest calibration error check and monitor
and record the temperature regularly (at least once each 5 minutes) during the sample collection period. If at any time during sampling the NO cell temperature is 85°F or greater and has increased or decreased by more than 5°F since the pretest calibration, stop sampling immediately and conduct a post test calibration error check per Section 7.6, re-zero the analyzer, and then conduct another pretest calibration error check before continuing.

7.5 Sample Collection. Position the sampling probe at the first measurement point and begin sampling at the same rate used during the calibration error check. Maintain constant rate sampling (i.e. ±10 percent of the analyzer flow rate value used in section 7.3.2) during the entire test run. Sample for an equal period of time at each test point. Sample the stack gas for at least twice the response time or the period of the stability time, whichever is greater, before collecting test data at each point. If recording emission data manually, record concentration values at least once each minute. If a computer or the analyzer record data automatically, the concentration data must be recorded either (1) at least once each minute, or (2) as a block average for the test run using values sampled at least once each minute. Do not break any seals in the sample handling system until after the post test calibration error test (this includes opening the moisture removal system to drain condensate).

7.6 Post Test Calibration Error Check. Immediately after the test run or set of test runs conduct span and zero calibration error checks using the procedure in Section 7.3. Conduct the calibration error check at the sampling location. Make no changes to the sampling system or analyzer calibration until all of the calibration error test results have been recorded. If the zero or span calibration error exceeds the specifications in Section 4 then all test data collected since the previous calibration error check are invalid. If the sampling system is disassembled or the analyzer calibration is adjusted, repeat the calibration error check before conducting the next test or test run.
7.7 **Interference Verification.** The tester shall review the results of the post test calibrations and compare them to the results of the most recent interference test. Use the post test calibration results and average emission concentrations for the test to calculate interference responses ($I_{W}$ and $I_{CO}$) using the procedure in section 6.3. If an interference response exceeds 5%, all emission test results since the last successful interference test for that compound are invalid.

7.8 **Re-Zero.** At least once every three hours or each time the analyzer sampling location changes, recalibrate the analyzer at the zero level according to the manufacturer's instructions and conduct a pretest calibration error test before resuming sampling. If the analyzer is capable of reporting negative concentration data (at least 5% of the span gas below zero), then the tester is not required to re-zero the analyzer.

8. **CALIBRATION CORRECTIONS**

8.1 The tester may choose to correct the emissions data for a test run using the pretest and post test calibration error results. Use the following formula to make the corrections:

\[
C_{\text{GAS}} = (C_R - C_o) \frac{C_{MA}}{C_M - C_o}
\]

where:

- $C_{\text{GAS}}$ = corrected flue gas concentration
- $C_R$ = flue gas concentration indicated by gas analyzer
- $C_o$ = average of initial and final zero checks
- $C_M$ = average of initial and final span checks
- $C_{MA}$ = actual concentration of span gas

9. **BIBLIOGRAPHY**

FIGURE 1.
CALIBRATION SYSTEM SCHEMATIC
FIGURE 2.
LINEARITY CHECK DATA SHEET

Date____________________ Analyst____________________

Analyzer Manufacturer/Model No.____________________
Analyzer Serial Number____________________

<table>
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<th>Calibration Gas Conc.</th>
<th>ppm NO Response</th>
<th>ppm NO₂ Response</th>
<th>ppm CO Response</th>
<th>% Oxygen Response</th>
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FIGURE 3.
STABILITY CHECK DATA SHEET

Date____________________ Analyst____________________

Analyzer Manufacturer/Model No. __________________________
Analyzer Serial Number ________________________________
Pollutant Channel__________ Span Gas Concentration__________

<table>
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<th>Elapsed Time (Minutes)</th>
<th>Analyzer Response</th>
<th>Elapsed Time (Cont.)</th>
<th>Analyzer Response</th>
<th>Elapsed Time (Cont.)</th>
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For 30-minute stability period: _______ maximum _______ minimum

For 15-minute stability period: _______ maximum _______ minimum

Maximum Deviation = 100 x (max. - min.)/span gas conc. = ________

Stability Time__________
FIGURE 4.
CALIBRATION ERROR CHECK DATA SHEET

Emission Source _______________________________________________________________________

Emission Test Run Number(s)_________ Start Test Period_______
Stop Test Period_______

Date__________
Analyst__________________________

Analyzer Manufacturer/Model No.___________________________________________________________
Analyzer Serial Number_______________________________________________________________

Pretest Calibration Error Check Results:

Initial NO cell temperature __________

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Post Test Calibration Error Check Results:

Final NO cell temperature __________

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