Standard Operation Procedures for NO-NO_2-NO_X

In Use By

Polk County Air Quality
Ambient Air Monitoring Personnel

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Polk County Air Quality Air Pollution Monitoring Specialist — *Date* 10/15/13

SIGNATURES/APPROVALS

Brent Blanchard
Polk County Air Quality Assurance Officer — *Date* 10/17/2013

POLK COUNTY AIR QUALITY DIVISION
STANDARD OPERATING PROCEDURE MANUAL FOR THERMO SCIENTIFIC $NO-NO_2-NO_X$ MONITORING

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2 .0 STANDARD OPERATING PROCEDURE FOR $NO-NO_2-NO_X$

2.1 Purpose

To establish a standard operating procedure (SOP) manual for the Polk County Air Quality Division personnel concerning the setup, operation, bi-weekly zero/precision/span checks, calibrations, audits and maintenance of $NO-NO_2-NO_X$ monitors maintained and operated by Polk County staff. This SOP is intended for individuals responsible for collecting ambient air monitoring data supported by the Polk County Air Quality Division.

2.2 Scope

These procedures are to be used by Polk County Air Quality Division personnel. The objective of this SOP is to familiarize the station operator with procedures used in the collection of air monitoring data. The accuracy of the data obtained from any instrument depends upon the instrument's performance and the operator's skill. It is important that the station operator become familiar with both this SOP as well as the manufacturer's instruction manual in order to achieve a high level of data quality. This SOP is to be used as an outline and is not intended to replace the equipment manufacturer's manual or procedures. This SOP describes the proper procedures for the setup, operation, weekly zero/precision/span checks, calibrations, audits and maintenance of NO_X equipment operated by Polk County Air Quality personnel.

2.3 References

2.3.1 Thermo Scientific, Model 42*i*, Chemiluminescence $NO-NO_2-NO_x$ Analyzer, Instruction Manual. Part Number 101350-00, December 28, 2007.

2.3.2 Thermo Electron Corporation, Model 146*i*, Dynamic Gas Calibrator, Instruction Manual. Part Number 102482-00, January 22, 2006.

2.3.3 EPA Quality Assurance Guidance Document 2.3, Reference Method for the Determination of Nitrogen Dioxide in the Atmosphere (Chemiluminescence), February, 2002, Draft.

2.3.4 EPA-600/4-75-003 December 1975, Technical Assistance Document for the Chemiluminescence Measurement of Nitrogen Dioxide.

2.3.5 EPA-600/R-92-121 September 1997b, EPA Traceability Protocol Assay and Certification of Gaseous Calibration Standards.

2.3.6 EPA-454/B-13-003, May 2013. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II. Ambient Air Quality Monitoring Program.

2.3.7 40 Code of Federal Regulations (CFR) Part 50, Appendix F, Measurement Principle and Calibration Procedure for the Measurement of NO_X in the Atmosphere.

2.3.8 40 Code of Federal Regulations (CFR) Part 58, Appendix A, Quality Assurance Requirements for State and Local Air Monitoring Stations (Slams).

2.3.9 40 Code of Federal Regulations (CFR) Part 58, Appendix D, Network Design for State and Local Air Monitoring Stations (SLAMS), National Air Monitoring Stations (NAMS), and Photochemical Assessement Monitoring Stations (PAMS).

2.3.10 40 Code of Federal Regulations (CFR) Part 58, Appendix E, Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring.

2.4 Introduction

The U.S. Environmental Protection Agency (EPA) has determined nitrogen dioxide (NO_2) is a health and environmental concern. For this reason, National Ambient Air Quality Standards (NAAQS) have been established for NO_2. As a result, an air monitoring network to monitor for NO_2 was established in the Des Moines Metropolitan Statistical Area (MSA) and is maintained by Polk County Air Quality Division, Iowa.

Measurements of nitrogen dioxide (NO_2) in ambient air are based on the principle that nitric oxide (NO) and ozone (O_3) react to produce a characteristic luminescence with an intensity linearly proportional to the NO concentration. Infrared light emission results when electronically excited NO_2 molecules decay to lower energy states:

$$NO + O_3 \rightarrow NO_2 + O_2 + h\nu$$

Ambient air is drawn into the $NO\text{-}NO_2\text{-}NO_x$ sample inlet. The sample flows into a solenoid valve which splits the flow between the NO and NO_x mode. NO_x mode converts NO_2 to NO (measuring total NO_x) while the NO mode makes no changes to the air stream and measures NO only. Samples from both modes flow to a reaction chamber where they are alternately introduced to react with dry air passed through an ozonator to provide a source of O_3. The O_3 reacts with the NO present in each sample producing electronically excited NO_2 molecules. A photomultiplier tube housed in a thermoelectric cooler detects the NO_2 luminescence and converts it to an electrical signal, which is then sent to the analyzer's front panel display and analog outputs. The concentration of NO and NO_x are both measured with NO_2 concentration calculated by difference. ($NO_x - NO = NO_2$).

The dynamic parameter requirement ensures that the $NO\text{-}O_3$ reaction has been completed. The dynamic parameter conditions are met for any reasonable NO flow (12.5–100 sccm) and [NO]STD (40-60 ppm). If the NO concentration of the stock gas cylinder, or flow through the NO MFC in the calibrator falls outside of these ranges, a calculation will be done to ensure that the residence time is ≤ 2 minutes and the dynamic parameter is ≥ 2.75 ppm-min, in accordance with formulas given to the analyzer operator's manual. Section 8-4, Thermo Scientific 146*i* Instruction Manual.

2.5 Health and Safety Warning/Precautions

Only properly trained personnel should perform 42*i* testing, installation, operation, maintenance and calibration procedures. As with all monitoring equipment, precautions should be taken when working around electricity, power tools and above ground elevations.

Cylinder gases are used in tandem with Mass Flow Control (MFC) calibrators for the 42*i*. Gas cylinders can sometimes contain pressures as high as 2000 pounds per square inch (psi). Handling of cylinders must be done in a safe manner. If a cylinder is accidentally dropped and valve breaks off, the cylinder can become explosive or a projectile.

Transportation of cylinders is regulated by the Department of Transportation (DOT). It is strongly recommended that all agencies contact the DOT or Highway Patrol to learn the most recent regulations concerning transport of cylinders. It is possible to blend other compounds with CO cylinder gas. In this case, it is recommended that MSDS for all compounds be made available to all staff that use and handle the cylinders or permeation tubes.

2.6 Sampler Setup

2.6.1 Site Selection

In selecting a location to measure for NO_2, it is important to understand that the reactive oxides of nitrogen in the atmosphere are primarily nitric oxide (NO) and nitrogen dioxide (NO_2), known together as NO_x. During the daytime, there is a rapid interconversion of NO and NO_2. NO emitted into the air is converted to NO_2 by photochemical reactions promoted by sunlight eventually creating ozone downwind of the sources. The main sources of NO_x are motor vehicles, power plants, industry, and

outdoor burning. It is recommended that the station be located downwind of the expected point of maximum NO_x to allow more time for the formation of NO_2.

For more detailed information concerning site selection for NO-NO_2-NO_x monitoring, refer to the 40 Code of Federal Regulations (CFR), Part 58, Appendix D, or the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Section 6.2, entitled "Monitoring Site Location."

2.6.1.1 The Monitoring Station

The structure housing the monitoring equipment may consist of a trailer, a room in a building, or a shelter designed specifically for air monitoring purposes. A clean, dry, secure and temperature controlled space is required so that the sampling equipment can operate properly.

Careful thought and planning is required in locating a monitoring station. The individual responsible for the installation must consider:

- Proximity to the nearest power source. A 120 VAC source is required for the operation of the NO-NO_2-NO_x monitoring equipment.
- The space where the equipment is housed must maintain a temperature range of 20-30 degrees Celsius. This usually requires the need for an air conditioner and a heater controlled by a thermostat.
- The accessibility of the equipment to the operator. The operator must be able to safely access the equipment during regular business hours.
- The security of the equipment. Monitoring instruments are expensive. They must be placed in a location where security can be assured.
- Contracts for rental of space or power. Contracts need to be signed with the owner of the property where the instruments are located.
- Telephone lines for data transmission to a central computer. A four-wire, dedicated 1200 baud (minimum) telephone data line is needed to access the Polk County Air Quality telemetry.
- Local building codes. In most cases, the contractor installing the power, structure, concrete, etc. know the local building codes.
- Dirty, dusty areas must be avoided.

2.6.1.2 Probe Placement

Once the location of the station has been identified, the individual responsible for the installation must be familiar with the criteria for locating the probe. The location of the sample probe is critical and individuals performing the installation must follow these specific guidelines:

- The inlet probe must be located between 3-15 meters about ground level.
- The inlet probe must be at least 1 meter vertically or horizontally away from any supporting structures.
- To avoid interference from obstructions, air flow must be unrestricted in an arc of at least 270 ° around the inlet probe, or 180° if the probe is on the side of a building.
- Trees can provide surfaces for NO_2 adsorption or reactions and obstruct wind flow. To reduce this possible interference, the inlet probe must be at least 10 meters from the drip line of the trees.
- When siting NO_2 analyzers for neighborhood and urban scale monitoring, it is important to minimize interferences from automotive sources. Minimum

separation distance between roadways and probes depends on the roadway's average daily traffic.

For more detailed information concerning site selection for NO-NO_2-NO_x monitoring, refer to the 40 Code of Federal Regulations (CFR), Part 58, Appendix E, or the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II.

2.6.2 Inspecting New Equipment

When shipment of the monitor is received, verify that the package contents are complete as ordered. Inspect the instrument for external physical damage due to shipping, such as scratched or dented panel surfaces and broken knobs or connectors.

Remove the instrument cover and all interior foam packing and save (in case future shipments of the instrumentation are needed). Make note of how the foam packing was installed.

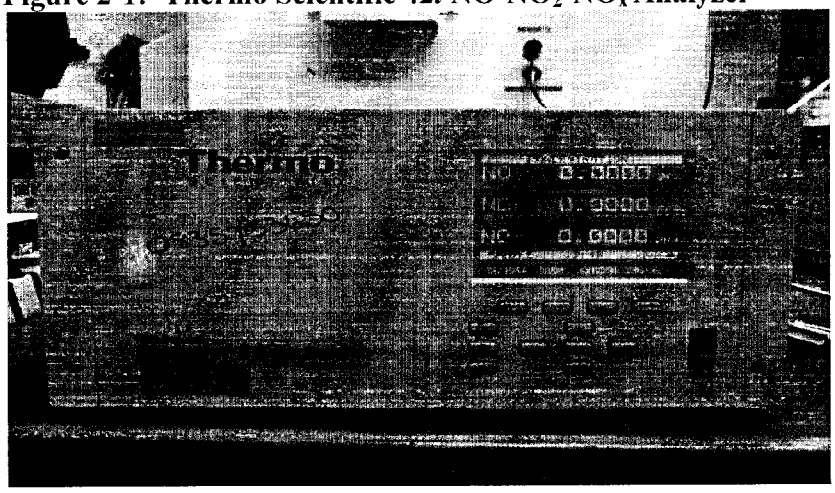
Inspect the interior of the instrument for damage, such as broken components or loose circuit boards. Make sure that all of the circuit boards are completely secured. Loose boards could short out the motherboard. If no damage is evident, the monitor is ready for calibration, installation and operation. If any damage due to shipping is observed contact Thermo Scientific at 1-866-282-0430 for instructions on how to proceed.

If you discover that the instrument was damaged during shipping and it becomes necessary to return it to the manufacturer, repack it in the same way it was delivered.

2.6.3 Equipment Installation

Polk County Air Quality Division will use the Thermo Electron Corporation Model 42*i* Chemiluminescence NO-NO_2-NO_x Analyzer displayed in Figure 2-1 for sampling nitrogen dioxide. Installation of the 42*i* consists of connecting the sample tubing to the sample gas inlet fitting and connecting the primary power and the data logger device. The sampler inlet line connection should be made with ¼ -inch outer diameter Teflon tubing.

Figure 2-1: Thermo Scientific 42*i* NO-NO_2-NO_x Analyzer



The entrance of the sampling system must have provision for a water drop-out or other means of ensuring that rain cannot enter the system. Place this water drop-out as far as possible from any sources that could contaminate the sample.

Because the analyzer is an optical instrument, it is possible that particulate in the gas sample could interfere with the NO-NO_2-NO_X readings. Although, the sampling/referencing cyclic operation of the instrument is designed to eliminate such interference. In order to avoid frequent cleaning of the optics and flow handling components, installation of a Teflon filter between the ambient sample line and the sample port of the analyzer must be done before the operation of the analyzer. A 0.5-micron Teflon filter will not degrade the NO_2 concentration. However, if particulate matter builds up on the filter, the particulate matter will destroy some of the NO_2 in the sample.

Since the instrument's exhaust consists of ambient air with some NO_2 removed, ensure that the exhaust cannot re-enter the sample system.

Install the monitor's electrical connections as indicated in the manual.

The power backup, data acquisition equipment, and any monitoring equipment, calibration equipment, or other ancillary equipment should be installed according to information supplied in the appropriate manuals.

2.6.4 Programming the Equipment

When the instrument is first turned on, the exhaust fan will start and the Power-Up and Self-Test screens will be displayed. These screens will be displayed until the instrument has completed its warm up and self-checks. Allow 30 minutes for the instrument to stabilize.

After the warm-up period the Run Screen, or Normal Operating Screen, is displayed. The Run Screen displays the NO-NO_2-NO_X concentrations. Press the MENU button to access the Main Menu, which contains a list of submenus. Instrument parameters and features are divided into the submenus according to their function. Use the ↑ **or** ↓ buttons to move the cursor to each submenu.

2.6.4.1 Range and Units

The Range menu defines the concentration range of the analog outputs. Polk County Air Quality Department will use the "Single Range" option with the selected range of 0 – 250 ppb for the analyzer. To set the range for the instrument, press the **MENU** button to access the Main Menu. Press the ↓ button until the cursor is on "NO Range." Press **ENTER** to display the NO Range Menu, and select **RANGE**. Use the ↑ **or** ↓ buttons to scroll through the preset ranges. Select "0.500" and press **ENTER**. Press **MENU** to return to the Range Menu. Repeat procedures to set NO_2 Range and NO_X Range.

The Gas Units Screen defines how the NO-NO_2-NO_X concentration reading is expressed. From the Range Menu, select **GAS UNITS** to display the Gas Units screen. Use the ↓ button to select "PPB" and press **ENTER**. Press **MENU** twice to return to the Run Screen.

2.6.4.2 Averaging Time

The averaging time defines a time period (1 to 300 seconds) during which NO-NO_2-NO_X measurements are taken. The average concentration of the readings is calculated for that time period. The Polk County Air Quality Department will use the default setting of 10 seconds as

the averaging time for the collection of data. An averaging time of 300 seconds will be used during calibrations, audits, and zero/precision/span checks.

From the Main Menu, use the ↓ button to scroll to **AVERAGING TIME** and press **ENTER**. From the Averaging Time Screen, use the ↑ **or** ↓ buttons to scroll through the preset ranges. Press **ENTER** to select the desired averaging time. Press **RUN** to return to the Run Screen.

2.6.4.3 Temperature Compensation

Temperature compensation corrects for any changes to the instrument's output signal due to variations in internal instrument temperature. When the temperature compensation is off, the first line of the display shows the factory standard temperature of 30°C. The Polk County Air Quality Department will run the NO-NO_2-NO_X analyzer with the temperature compensation set to **ON**.

From the Main Menu, use the ↓ button to scroll to **INSTRUMENT CONTROLS** and press **ENTER**. From the Instrument Controls Screen, use the ↓ button to scroll to **TEMPERATURE COMPENSATION**, and press **ENTER**. Press **ENTER** to toggle the temperature compensation on or off. Press **RUN** to return to the Run Screen.

2.6.4.4 Pressure Compensation

Pressure compensation corrects for any changes to the instrument's output signal due to variation in the reaction chamber pressure. When the pressure compensation is off, the first line display shows the factory standard pressure of 150 mmHg. Polk County Air Quality Department will run the NO-NO_2-NO_X analyzer with the pressure compensation set to **ON**.

From the Main Menu, use the ↓ button to scroll to **INSTRUMENT CONTROLS** and press **ENTER**. From the Instrument Controls Screen, use the ↓ button to scroll to **PRESSURE COMPENSATION**, and press **ENTER**. Press **ENTER** to toggle the pressure compendsion on or off. Press **RUN** to return to the Run Screen.

2.6.4.5 Baud Rate

The Baud Rate Screen is used to set the RS-232 interface baud rate. The Polk County Air Quality Department will use a baud rate of 9600.

From the Main Menu, use the ↓ button to scroll to **INSTRUMENT CONTROLS** and press **ENTER**. From the Instrument Controls Screen, use the ↓ button to scroll to **COMMUNICATION SETTINGS** and press **ENTER**. From the Communication Settings Screen, use the ↓ button to scroll to **BAUD RATE**, and press **ENTER**. Use the ↑ **or** ↓ buttons to scroll through the preset rates. Press **ENTER** to select the desired baud rate. Press **RUN** to return to the Run Screen.

2.6.4.6 Clock Set

To set the correct time and date on the instrument, press **MENU** to return to the Main Menu. Use the ↓ button to scroll to **INSTRUMENT CONTROLS** and press **ENTER.** Use the ↓ buttons to scroll to **DATE/TIME** and press **ENTER**. The date and time should be set to the data logger time. Use the → button to select: year, month, day, hour, minutes, or seconds. Use the ↑ **or** ↓ buttons to increase/decrease the desired value. Set the appropriate date and time and press

ENTER. Press **RUN** to return to the Run Screen. The instrument is now set with the appropriate time, date, full scale range and units.

NOTE: The clock should be checked at each site visit and adjusted if off from the data logger by more than 2 minutes.

NOTE: It is recommended that you allow the 42*i* to warm up for 24-hours before you attempt checks or calibration.

2.7 Standards

A cylinder containing 10 to 20 ppm NO in N_2 with less than 1 ppb NO_2 is used as the concentration standard. The cylinder must be traceable to a National Institute of Standards and Technology (NIST) NO in N_2 Standard Reference Material or NO_2 Standard Reference Material. Procedures for certifying the NO cylinder (working standard) against a NIST traceable NO or NO_2 standard and for determining the amount of NO_2 impurity are given in EPA Publication NO. EPA-006/4-75-003, "Technical Assistance Document for the Chemiluminescence Measurement of Nitrogen Dioxide." In addition, the procedure for the certification of a NO working standard against a NIST traceable NO standard and determination of the amount of NO_2 impurity in the working standard is reproduced here. The cylinder should be recertified on a regular basis as determined by the local quality control program.

The gas certification process is quite rigorous. However, it is still possible to receive a cylinder gas out of specification. At the request of the IDNR, Polk County Air Quality will verify that a new cylinder gas is within specification before it is put into use. Immediately prior to removing an old cylinder, perform a zero/precision/ span check, making sure that relevant criteria are met. Change to the new cylinder, and update the Thermo 146*i* with the new stock gas concentration. Adjust the 146*i* to produce a target concentration equivalent to the precision level last used with the old cylinder. If the instrument reading is within 4% of the expected value, proceed to calibrate the analyzer with the new stock cylinder. If it is not within 4%, investigate and resolve the disparity prior to using the new cylinder.

Operators will be required to replace the NO cylinder when the cylinder pressure drops below 200 PSI. This will involve removing the regulator on the depleted cylinder and installing the regulator on a replacement cylinder.

Precautions must be taken to remove "dead" pockets of contaminants which are created within the regulator whenever it is removed from the cylinder. This problem can be minimized by carefully evacuating the regulator (also known as purging) after it is connected to the cylinder. Air trapped in the regulator can result in the NO converting to NO_2 within the regulator resulting in errors during calibration. Better results will be achieved by alternately pressurizing and depressurizing the regulator once it has been attached to the cylinder.

Contamination with even a small amount of moisture from back diffusion can cause the NO concentration to become unstable. The lower the cylinder concentration, the more susceptible it is to any contamination from "abuse" in the field. The best way to ensure low concentration cylinders are not contaminated by back diffusion is to make sure whenever the cylinder valve is open, there is gas flow out of the cylinder. If this procedure is always applied, it is impossible for any air or other contaminants to enter the cylinder, and the practice of repeated vacuum purging of regulators is not necessary.

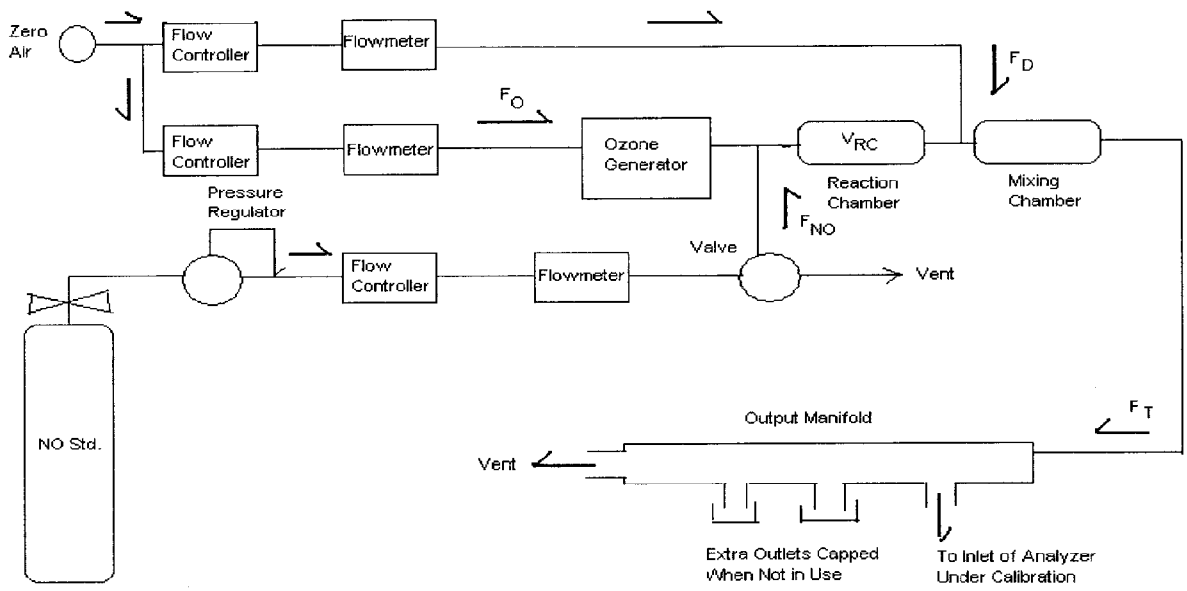
Polk County addresses this issue by:

- The cylinder utilized is only used for this site and is not removed until expiration of the certificate.
- The gas dilution system is fixed at this site and is not used at any other site so is not moved unless for maintenance or re-certification.
- The regulator will continuously be kept pressurized with cylinder gas.

2.8 Qualification, Certification and Recertification

Gas dilution in the Model 146*i* Multi-Gas Calibrator is achieved by utilizing two mass flow controllers. One is a high flow controller (typically 20 slm full scale) to govern the diluting zero air. The other controller is for low flow (typically 100 sccm) and governs the flow of the gas to be diluted. The Mass Flow Controller must be verified once every quarter in order to recertify the 146*i* as a working transfer/audit standard. See Polk County Air Quality SOP Section 26 for calibration instructions of the 146*i* Mass Flow Controllers.

Figure 2-2: Gas Phase Titration System



2.9 Calibrations

The Thermo Scientific Model 42*i* NO-NO_2-NO_x monitor is calibrated whenever the following occurs: installation of a new monitor, replacement of broken parts, when a zero/precision/span check fail to meet acceptance criteria limits, after 6 months has expired since the last calibration, or at the operator's discretion.

The Purpose of the calibration is to determine the NO, NO_2 and NO_x relationship between the analyzer and the true NO and NO_2 concentrations. This procedure involves the gas phase titration (GPT) of a NO standard with O_3 to produce NO_2. This method is based upon the rapid gas phase reaction between NO and O_3 to produce stoichiometric quantities of NO_2.

$$NO + O_3 \rightarrow NO_2 + O_2$$

The calibration check is a quality control procedure used to verify that the air monitoring system is operating properly. The check involves comparing the response of the station analyzer to NO-NO_2-NO_x concentrations generated by the station gas calibration system.

Using linear regression, a calibration relationship is determined using the indicated values of the analyzer and the actual values from the calibrator. The calibration data is saved for use as a point of reference for subsequent calibrations.

2.9.1 Calibration Procedures for the 42*i* using the 146*i* Calibrator

2.9.1.1 Pre-Calibration

Change the averaging time to 300 seconds. From the Main Menu, use the ↓ button to scroll to **AVERAGING TIME** and press **ENTER**. From the Averaging Time Screen, use the ↑ **or** ↓ buttons to scroll through the preset ranges. Press **ENTER** to select an averaging time of 300 seconds. Press **RUN** to return to the Main Screen.

Make sure that the ozonator in the 42*i* is on. Press the front panel **MENU** button on the 42*i* site analyzer to display the Main Menu. Use the arrow keys to select **INSTRUMENT CONTROLS** and press **ENTER**. Scroll to **OZONATOR** and press enter. Press **ENTER** again to turn the ozonator on.

2.9.1.2 Calibration of NO/NO_x

The GPT requires the use of the NO/NO_x channel of the analyzer to determine the amount of NO_2 generated by titration. Therefore, it is necessary to calibrate and determine the linearity of the NO/NO_x channels before proceeding with the NO_2 calibration.

On the Main Screen (or Standby Screen) of the 146*i* Calibrator, press the **MENU** button to access the Main Menu Screen. Scroll to **OPERATION** and press **ENTER**. Use the ← and → buttons to select desired **GAS** and press **ENTER**. Use the ↓ button to scroll down to **SPAN**. Use the ← and → buttons to scroll to **ZERO** and press **ENTER** to put the 146*i* Calibrator in zero air mode. Allow the analyzer to sample zero gas for a minimum of 15 minutes until stable readings are obtained on the NO, NO_2, and NO_X channels.

Press the front panel **MENU** button on the 42*i* site analyzer to display the Main Menu. Use the arrow keys to select **CALIBRATION** and press **ENTER** to display the Calibration Menu.

Select **CALIBRATE NO BACKGROUND** and press **ENTER**. Press **ENTER** to set the NO reading to zero. Press the **MENU** button to return to the Calibration Menu.

Select **CALIBRATE NO_X BACKGROUND** and press **ENTER**. Press **ENTER** to set the NO_X reading to zero. Press the **MENU** button to return to the Calibration Menu.

Adjust the flow rate from the NO standard to generate an NO concentration of approximately 80% the upper range limit (URL) of 250 ppb (i.e. 200). Allow the site analyzer to stabilize for a minimum of 15 minutes and record all data logger readings from the NO, NO_2, and NO_X channels.

Press the front panel **MENU** button on the 42i site analyzer to display the Main Menu. Use the arrow keys to select **CALIBRATION** and press **ENTER**. Scroll to **CALIBRATE NO COEFFICIENT** and press **ENTER**. Use the ← and → buttons to move the cursor left and right. Use the ↓ or ↑ buttons to increment and decrement the digit to the expected NO concentration and press **ENTER**.

Repeat to calibrate the NO_X concentration.

NOTE: The NO and NO_X Background coefficients should be less that 15 ppb. The NO, NO_2 and NO_X span coefficients should always fall between 0.900 and 1.100. If outside that range, refer to Thermo Scientific's Instruction Manual Chaper 7 "Servicing" procedures for PMT Voltage Adjustment.

After the zero and 80% URL points have been set, determine at least 4 approximately evenly spaced points between zero and the 80% URL without further adjustment to the analyzer. SPAN 1 - 5 have been programmed for calibrations. SPAN 2 is set to 160 ppb, SPAN 3 is set to 120 ppb, SPAN 4 is set to 80 ppb, and SPAN 5 is set to 40 ppb.

Allow the site analyzer to stabilize for a minimum of 15 minutes at each SPAN Point and record all data logger readings from the NO, NO_2, and NO_X channels.

Repeat for SPAN 2 - 5, respectively. Record all information on the Calibration Field Sheet, Form 1, Appendix A.

2.9.1.3 Calibration of NO_2

On the main screen of the 146*i* Calibrator, use the ↓ button to scroll to the third line. Use the ← and → buttons to scroll to **OZON MAN**.

Note: Check and make sure that the second line is set to 225 ppb. The NO_2 calibration must be performed using an NO_x concentration of 450 ppb.

Push the **MENU** button on the 146*i* Calibrator. Scroll to **OZONATOR SETUP** and press **ENTER**. Scroll to **MANUAL** and press **ENTER.** The ozone reading on the 146*i* Calibrator will be displayed as a percentage. Use the arrow keys to set the ozone level to 0%. When the analyzer responses stabilize, record the resultant data logger readings on the NO, NO_2, and NO_X channels. NO_2 should stabilize out at approximately 0 ppb. NO/NO_x should stabilize out at approximately 225 ppb. Record all information in the site log.

At this point the zero NO_2 reading has been performed. Press the **MENU** button on the Calibrator. Scroll to **OZONATOR** and press **ENTER**. Scroll to **MANUAL** and press **ENTER.** Use the arrow keys to adjust the ozonator percentage to obtain a NO_2 reading in the range of 400 ppb. Allow the site analyzer to stabilize for a minimum of 15 minutes and record all data logger readings from the NO, NO_2, and NO_X channels.

NO_2 is calibrated according to a direct comparison between the NO concentration obtained when NO_2 is set to zero and the NO concentration obtained at approximately 80% the URL of the uncalibrated NO_2 channel (i.e. 200).

$$NO_{2Actual} = (NO_{orig} - NO_{rem}) + NO_{2(imp)}$$

Where:
NO_{orig} = Original NO data logger reading, when NO_2 is set to zero
NO_{rem} = Final NO data logger reading when NO_2 is approx. 200 ppb
$NO_{2(imp)}$ = Conc. of NO_2 impurity in standard NO cylinder

The NO_2 impurities in the NO concentration standard are determined by:

$$NO_{2(imp)} = \frac{(F_{NO}\ (sccm))(\text{Impurities from cylinder gas (ppb)})}{F_O + F_D}$$

Where:
F_{NO} = NO Flow
F_O = Ozone Flow
F_D = Dilution Flow

Press the front panel **MENU** button on the 42i site analyzer to display the Main Menu. Use the arrow keys to select **CALIBRATION** and press **ENTER**. Scroll to **CALIBRATE NO_2 COEFFICIENT** and press **ENTER**. Use the ← and → buttons to move the cursor left and right. Use the ↓ or ↑ buttons to increment and decrement the digit to the expected NO_2 concentration. Press **ENTER** to calibrate the analyzer to the desired NO_2 concentration.

NOTE: If the analyzer calculates a NO_2 span coefficient of less than 0.96, either the entered NO_2 concentration is incorrect, the converter is not being heated to the proper temperature, the instrument needs servicing, or the converter needs replacement or servicing.

After the zero and 80% URL points have been set, determine at least 4 approximately evenly spaced points between zero and the 80% URL without further adjustment to the analyzer. Record all information on the Calibration Field Sheet, Form 1, Appendix A.

Generate the regression analysis calculations by least squares the slope, intercept, and correlation coefficient of the site analyzer response versus the calibration standard concentrations, Form 2, Appendix A.

Record all information in the site log.

2.9.2 Converter Efficiency (NO_2)

During a calibration, the converter efficiency must be determined for each NO_2 level. The converter efficiency slope criteria of 0.96 to 1.04 explicitly applies to each level. Use the Converter Efficiency Spreadsheet to determine if the calibration passes, Form 3, Appendix A. See Appendix B for detailed instructions for using the Converter Efficiency Spreadsheet.

For more detailed information concerning calibration of a Thermo Scientific 42*i* NO-NO_2-NO_x analyzer, refer to the Quality Assurance Guidance Document 2.3, Reference Method for the Determination of Nitrogen Dioxide in the Atmosphere (Chemiluminescence), February, 2002, Draft; and the Instruction Manual, Model 42*i*, Cheimluminescence NO-NO_2-NO_x Analyzer.

2.10 Quality Control

2.10.1 Station Inspection

Before entering the station, the perimeter should be inspected for damage. Extreme weather conditions, neglect of station maintenance or vandalism may have resulted in damage to the site since the operator's last visit. Check that the sample probe is intact and has not been damaged.

Once the operator has entered the monitoring station, she/he should first:

- Check for any obvious analyzer malfunctions. For example, check to see that the equipment is running, the pumps are operating and the instrument is cycling properly.
- Note any unusual odors or noise. An unusual odor may indicate a point source of a pollutant or a strange new noise can indicate a malfunction in the equipment. These observations should be recorded in the station log book and may prove to be invaluable if the data is challenged.

The station operator is responsible for making several observations during the station inspection. Any of the above described observations must be thoroughly detailed in the site log book.

Once the initial inspection is made, the operator must proceed with a routine inspection and perform a zero/precision/span check on the NO-NO_2-NO_x analyzer.

2.10.2 Station Log Book

A station log book must be maintained at each monitoring site and should accurately reflect site operations. The log book will be identified with the station name, station number, date, time, operator, instrument identification, parameter, scale and units. All entries shall include the date, time, quality control checks, and maintenance on equipment, audits, equipment changes and missing or invalid data. Additional information should include: maintenance performed on the station, abnormal traffic patterns, nearby construction, or sample line cleaning.

Should the data be challenged, the information recorded in the log book is invaluable. A written record of observations concerning abnormal operations or localized occurrences is critical if a violation of ambient air standards were recorded during this period. Completed log books be maintained by Polk County Air Quality and will be archived for future reference.

2.10.3 Bi-Weekly Zero, Precision and Span Checks

The zero, precision and span check is a quality control procedure used to verify that the air monitoring system is operating properly. The check involves comparing the response of the station analyzer to NO/NO_x concentrations generated by the station 146*i* calibrator. The deviation between the "indicated" value of the analyzer and the "actual" or "true" value of the calibrator is then determined.

Zero air and NO/NO_x concentrations of approximately 20 ppb (the precision) and approximately 200 ppb (the span) are generated by the calibrator. Each concentration is measured by the calibrator and the site analyzer. Following the precision check, the deviation from the true NO/NO_x value is determined.

The following critical criteria require recalibration of the field analyzer and invalidation of the data unless there is compelling reason and justification not to do so: if the precision check results in a percent

difference ± 15%, or if the zero drift is ≥ 1.5 ppb, or if the span drift is ± 10%. Acceptance criteria can be found in the Measurement Quality Acceptance Tables from the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Appendix D. EPA-454/B-13-003, May 2013: http://www.epa.gov/ttnamti1/files/ambient/pm25/qa/QA-Handbook-Vol-II.pdf.

The zero/precision/span check must be performed once every fourteen days and is always performed in the same manner. Failure to perform and document zero/precision/span data within the required frequency and concentration will result in the invalidation of data. The operator is required to submit the completed field sheet to the Quality Assurance Officer for review. For more detailed procedures on, Zero, Precision and Span Checks refer to Section 2.14.4.

2.10.4 Data Acquisition and Telemetry

Data acquisition involves retrieval of the ambient air quality data from the data logger. The station operator has the primary responsibility for distinguishing valid measurements from indications caused by malfunctioning instruments or source interferences. The telemetry system can be used by a station operator to scan data transmitted from the monitoring station to a central location. This enables the operator to "call" the monitor site and examine the data recorded at the monitoring station (i.e. NO-NO_2-NO_x concentrations and station temperature). The station operator should be familiar with daily concentration variations (i.e. the times daily maximum concentrations occur and the interrelationship of ozone). By recognizing abnormal data, the operator is alerted that the instruments may not be operating properly and a station visit may be necessary. However, monitoring a station by telemetry is not to be substituted for the site visit.

In the event of an exceedance of the 1-hour NO_2 standard, data averages of no longer that 5 minutes will be critical in validating the data and investigating the cause of the exceedance. All data loggers are set up to collect 1 minute, 15 minute and 1 hour averages, with 1 minute being the "base average". The 15 minute and hourly averages are called "extended average #1 and #2, respectively. The 1 minute, 15 minute and 1 hour averages are polled and stored before the data is overwritten. For more specific instructions on data acquisition, see Polk County Air Quality AirVision SOP Section 18.

2.11 Equipment, Maintenance and Trouble Isolation

The equipment used to calibrate, audit, perform bi-weekly zero/precision/span checks and monitor NO_X concentrations in the network are the Thermo Scientific Models 42*i* and the 146*i* Calibrator, Teledyne 701H Zero Air Generator, and the Agilaire Model 8832 data logger. A flow controller with a flow rate regulated ± 2% of readings over 20-100% of its full-scale range as specified in 40 CFR, Pt. 50, App. F. Compressed cylinder gas with an EPA protocol Gas as specified in EPA – 600/R97/12.

2.11.1 Preventative Maintenance

Each instrument must be periodically examined and serviced to anticipate and prevent instrument failure. Scheduled maintenance on the instruments will prevent costly repairs and loss of data. The routine maintenance required on the analyzers by the station operator is minimal and outlined in the manufacturer's manual. By keeping track of the instrument responses from week to week, the operator can observe trends, which would alert the operator of a potential problem, and to correct the situation before the instrument fails.

2.11.1.1 Analyzer Leak Check

An analyzer leak check should be performed on a yearly basis, or as needed for trouble shooting purposes. To perform a system leak check on the analyzer, disconnect the sample line from the **SAMPLE INLET** on the back panel, and plug all fittings except the exhaust port.

Press the **MENU** button to display the Main Menu. Use the ↓ buttons to scroll to **DIAGNOSTICS** and press **ENTER**. From the Diagnostic Menu, use the ↓ buttons to scroll to **SAMPLE FLOW** and press **ENTER**. The flow reading should slowly drop to zero. Press the **MENU** button to return to the Diagnostics Menu. Use the ↓ buttons to scroll to **PRESSURE** and press **ENTER**. The pressure reading should drop below 250 mmHg. If the flow and pressure do not drop to their desired limits, there is a leak in the system. Check to see that all fittings are tight, and none of the input lines are cracked or broken.

2.11.1.2 Sample Line Leak Check

A sample line leak check should be performed on a yearly basis, and anytime new sample line is installed. Unscrew the sample line from the back of the analyzer, and connect to the vacuum pressure gauge. The sample line must be capped off on the top side of the roof before proceeding.

Turn vacuum pump on and wait for the pump to remove all air from the sample line. The vacuum gauge will indicate a steady reading of approximately 21 inches of Hg vacuum when the lines have been evacuated. The vacuum reading must be at least 15 inches of Hg for the leak check to take place.

Close the valve that is located between the vacuum gauge and the pump. Once the valve is closed record the reading from the gauge and start a timer. Shut the pump off and wait 1 minute. Record the reading from the vacuum gauge.

The acceptable leak rate for the sample lines is 0.5 inches of Hg for the 1 minute check. If the leak check fails, make sure all lines are connected securely to the vacuum gauge and the line is capped off the roof, and repeat the check. If a second leak check fails, look for the leak and/or replace sample line if needed.

2.11.1.3 Particulate Filter Changes

Two 0.5 micron Teflon filters housed in Teflon filter holders are located on the outside of the analyzer (refer to Figure 2-6). One is located on the rear panel of the 42*i* analyzer, the other is connected to the sample line. The filter should be checked every two weeks and changed when noticeably dirty. Slow response of the analyzer during the zero/precision/span check is an indication of a dirty filter or contaminants in the sample line. To replace the used filter, loosen the two halves of the filter holder and replace the dirty filter with a new filter.

Following a filter change, perform a high span check (400 ppb) for at least half an hour. This is to "condition" the new filter and serves as a check to see if there any leaks in the reassembled filter holder.

2.11.1.4 Cleaning the Fan Filters

Under normal use, the fan filters on the rear panel should be cleaned every six months. If the instrument is operated in excessively dirty surroundings, it may be necessary to clean the fan filters more frequently.

2.11.1.5 Cleaning the PMT Cooler Fins

The Photomultiplier tube (PMT) cooler fins should be inspected and cleaned once every six months. This assures optimal performance of the cooler.

Using clean pressurized air, blow off the cooler fins. Make sure that any particulate accumulation between the fins has been removed. If necessary, employ a small brush to remove residual particulate accumulation.

2.11.1.6 Replacing the Sample Lines

It is the operator's responsibility to maintain the sample lines. The sample lines must be replaced if the operator suspects a loss in NO-NO_2-NO_x concentrations due to contamination in the line, or every two years.

2.11.2 Trouble Isolation

The instruments in Polk County Air Quality's air monitoring network are very reliable. However, after a period of continuous use problems may occur. Leaks in the system, built up dirt and U.V. lamp failure are typical. After time, the operator should be able to quickly distinguish the symptoms and causes of equipment failure.

It is suggested that each station operator consult the Model 42*i*, Chemiluminescence NO-NO_2-NO_x Analyzer Instruction Manual and compile personal notes on troubleshooting as they gain experience with the Thermo Scientific. The operator is encouraged to contact Thermo Scientific technical support at 1-866-282-0430 when attempting any repairs.

2.11.3 Environmental Control for Monitoring Equipment

Instrument vibration should be reduced as much as possible. Use shock-absorbing feet for the monitor. Any pumps must be fitted with rubber feet to reduce vibration. All pumps connected to the analyzer should be connected using tubing that will prevent the transfer of vibrations back to the instrument and/or the instrument rack.

All instruments should be shielded from natural or artificial light.

Ensure constant voltage to surge protection devices and equipment.

Regulate the housing temperature between 15-35°C. Hourly temperature readings are collected by the polling computer during daily polling. Polk County will make every effort to operate the monitor in the 15-35°C range. Data will be investigated to determine the validity of the data for hourly concentrations where the shelter temperatures fall outside the range of (15-35°C). Data collected during temperatures outside of this range may be subject to invalidation, in accordance with the December 2008 Redbook's guidelines for operational criteria.

2.12 Quality Assurance

The audit schedule for SLAMS monitoring, is that each analyzer must be audited at least once per year. Polk County should audit 25 percent of their analyzers per quarter as specified in Section 3.2.2 of 40 CFR Part 58 Appendix A. However, it is Polk County Air Quality Division's policy to audit the NO-NO_2-NO_X analyzer once per quarter.

2.12.1 Direct Comparison Audit

Once during each calendar quarter, the designated Quality Assurance Officer utilizing the procedures and calculations specified in 40 CFR 58, Appendix A, "Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS)," the NO-NO_2-NO_x analyzer will be audited using a direct comparison.

An audit is an independent assessment of the accuracy of data generated by an ambient air analyzer. Independence is achieved by having the audit performed by an operator other then the one conducting the routine field measurements and by using audit standards, reference materials, and equipment different from those routinely used in monitoring. Proper implementation of an auditing program will ensure the integrity of the data and assess the accuracy of the data.

An audit consists of challenging the 42*i* continuous analyzer with known concentrations of NO-NO_2-NO_X within the measurement range of the analyzer. The 146*i* Multi-Gas Calibrators can be programmed to generate the desired NO-NO_2-NO_X audit concentrations, see Polk County Air Quality SOP Section 26.

Generate at least three of the following SLAMS consecutive audit concentrations: 310-600 ppb, 110-300 ppb, 6-100 ppb, 3-5 ppb, and 0.02-2 ppb. The audit levels selected should represent 80 percent of the ambient concentrations measured by the analyzer. Allow each audit concentration to stabilize for a minimum of 15 minutes and record all data logger readings from the NO, NO_2, and NO_X channels. The results are recorded on the Quarterly Audit Sheet, Form 4, Appendix A. The NO_2 Converter Efficiency should also be determined during an audit. Use the Converter Efficiency Spreadsheet to determine if it passes, Form 5, Appendix A. See Appendix B for detailed instructions for using the Converter Efficiency Spreadsheet.

If the percent difference is outside ±15% or if the absolute value of the zero reading is greater than 1.5 ppb, and there is reason to believe the results are incorrect or not representative of the analyzers performance, the cause for the error must be investigated and corrected. An investigation initiated by audit results that are outside the above limits may require the invalidation of data. Record all information in the site log.

NOTE: For more detailed information concerning audit performances for NO-NO_2-NO_X monitoring, refer to the "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II" or 40 CFR 58.

2.12.2 Performance Evaluation Audit

Agency staff are sent an audit device, such as for the National Performance Evaluation Program (NPEP). The agency staff does not know the NO-NO_2-NO_x concentrations produced by the audit equipment. Responses of the on-site analyzer are then compared against those of the generator and a linear regression is calculated.

The Polk County Air Quality Department will participate in the USEPA performance audit program for NO-NO_2-NO_x and in all other EPA audit programs, which may arise in the future. According to the May 2013 Redbook, audit results must have a mean absolute difference of $\leq$ 15%. A review of the monitoring system shall be conducted if the results are above this, which may result with the invalidation of data. These blind audits will indicate any deficiency in the monitoring system in respect to precision and accuracy, calibrations and maintenance.

2.13 Data Quality Assessment

For each calendar quarter and year, Polk County Air Quality will prepare data precision, accuracy and completeness reports for the Iowa Department of Natural Resources (Iowa DNR) and EPA-Region 7 in accordance with the current Letter of Agreement (LOA).

2.13.1 Precision

Precision is defined as the measure of agreement among individual measurements of the same property taken under the same conditions. Precision is assessed from checks that are performed at least once every two weeks (see Section 3.14). Calculations to assess precision are given below and should be used to assess precision on a quarterly basis. It is recommended that the NO-NO_2-NO_x analyzer has a 90 percent confidence limit of coefficient of variation for precision of $\leq$15 %, and for bias as an upper 95 percent confidence limit for the absolute bias of 15%. The goal for acceptable measurement uncertainty is defined for precision, as an upper 90 percent confidence limit for the coefficient of variation (CV) of 15 percent; and for bias as an upper 95 percent confidence limit for the absolute bias of 15 percent.

The precision will be evaluated and reported employing the frequencies, procedures and calculations in 40 CFR Part 58, Appendix A, "Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS)".

2.13.2 Accuracy

The Polk County Air Quality Program participates in the USEPA performance audit program for NO_2 and in all other EPA audit programs, which may arise in the future. Using results from the performance audits and the calculations specified in 40 CFR 58, Appendix A, "Quality Assurance Requirements for State and Local Air Monitoring Stations". The accuracy will be evaluated and reported.

2.13.3 Data Completeness

The completeness of the data will be determined for each monitoring instrument and expressed as a percentage. Percent valid data will be a gauge of the amount of valid data obtained from the monitoring instrument, compared to the amount expected under ideal conditions (24 hours per day, 365 days per year). Exceptions will be made for analyzers with a seasonal sampling period, which were not installed at the beginning, or which were discontinued prior to the end of any reporting period for calculation purposes.

2.14 Procedures for Bi-Weekly Verifications

New station operators will be provided with on-site training by an experienced operator before they operate a station on their own. The following procedures are intended to assist the operator in performing and documenting monitoring procedures. Monitoring personnel must become familiar with the Operating and Maintenance Manuals for Thermo Scientific Model 42*i*, Chemiluminescence NO-NO_2-NO_x Analyzer, Instruction Manual, and Model 146*i*, Dynamic Gas Calibration System, Instruction Manual.

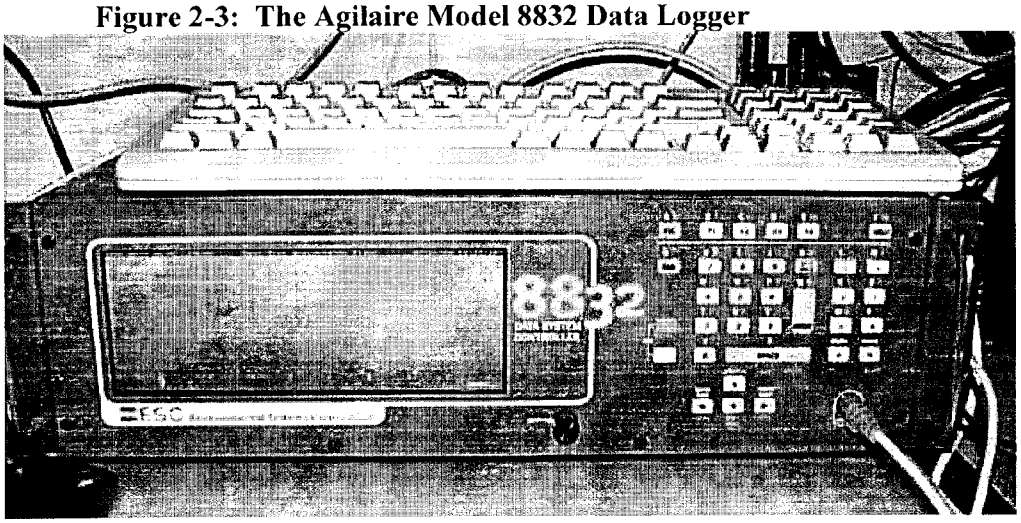
2.14.1 Maintenance Mode for Agilaire Model 8832 Data Loggers

Figure 2-3 displays the Agilaire Model 8832 data logger used for data collection. During any maintenance, zero/precision/span checks, calibrations, or site visit the data logger must be placed into maintenance mode. This mode flags hourly data with an (M), indicating the analyzer is in the process of being checked by the technician. Scroll down to **LOGIN/SET USER LEVEL** and press **ENTER.** Login to the data logger using the appropriate password and press **ENTER.** Use the arrow keys to select Configuration Menu and press **ENTER.** Use the arrow keys to select **CONFIGURE (DATA) CHANNEL** and press **ENTER.** Use the arrow keys to select **PUT CHANNEL IN MAINTENANCE MODE** and press **ENTER.** Select the correct pollutant channel and press **ENTER.** Repeat procedures for all appropriate channels. Proceed with maintenance, bi-weekly precision/span check, or calibration of the analyzer.

2.14.2 Remote Mode

Press the escape button (Esc) until the main menu is reached. Take the data logger out of maintenance mode when the task has been completed. Use the arrow keys to select Configuration Menu and press **ENTER.** Use the arrow keys to select **CONFIGURE DATA CHANNELS** and press **ENTER.** Use the arrow keys to select **TAKE CHANNEL OUT OF MAINTENANCE** and press **ENTER.** Repeat procedures to take all channels out of maintenance mode. Press the (Esc) button until the main menu is reached. Use the arrow keys to select **LOG OUT** and press **ENTER.** Record all information on tasks performed in the site log.

Figure 2-3: The Agilaire Model 8832 Data Logger



2.14.3 Maintenance and Diagnostics Check

Check and record monitor readings on the data logger. Check and record any alarms on the analyzer. If alarms are present, check field sheet for out-of-control limits and perform any necessary maintenance. See Model 42*i* Instruction Manual, Chapter 7-Servicing, and Section 2.11.1 Preventative Maintenance. Record any maintenance performed in the site log.

Check and record the calibration factors. Press **MENU** and scroll to **CALIBRATION FACTORS** and press **ENTER**. Record the background coefficients for NO and NO_x. Scroll down and record span coefficients for NO, NO_2, and NO_x.

Check the Teflon particulate filters and change as needed. If particulate matter builds up on the filter, the particulate matter will destroy some of the NO_2 in the sample.

Write down any maintenance that was performed on the instrument or additional comments that may affect the air monitoring system. Examples include:

- replacement of UV lamps, pumps or tubing
- repairs or maintenance made to or around the shelter
- abnormal localized occurrences nearby
- suggestions for improvements to the system
- supplies that are needed
- checks or audits that were performed

Table 2-1 Diagnostic Checks

Check	Explanation
Voltages	The DC power supply voltages
Temperatures	The internal instrument and chamber temperatures
Pressure	The reaction chamber pressure
Flow	The sample flow rate

2.14.4 Bi-Weekly Zero, Precision and Span Checks

2.14.4.1 Zero Air Check

The zero air must be free of contaminants that could cause a detectable response on the 42*i* analyzer. Polk County Air Quality Division uses the Teledyne 701H air generator with charcoal, Purafil®, and hydrocarbon scrubbers for the source of zero air. For more information on zero air scrubbers, see Polk County Air Quality SOP Section 25 for instructions for the Zero Air Module Model 701/701H.

The external zero air generator is connected to the zero air port on the back of the 146*i* Calibrator. The 146*i* Calibrator vent port is connected to the 42*i* site analyzer's sample port.

On the Main Screen (or Standby Screen) of the 146*i* Calibrator, press the **MENU** button to access the Main Menu Screen. Scroll to **OPERATION** and press **ENTER**. Use the ← and → buttons to select desired **GAS** and press **ENTER**. Use the ↓ button to scroll down to **SPAN**. Use the ← and → buttons to scroll to **ZERO** and press **ENTER** to put the 146*i* Calibrator in zero air mode. Allow the site analyzer to stabilize for a minimum of 15 minutes and record all

data logger readings from the NO, NO_2, and NO_X channels on the Verification Field Sheet, Form 6, Appendix A.

Polk County Air Quality uses the Agilaire Model 8832 data loggers for data recording. The values indicated on the data logger are the values that are being transmitted to, and recorded by the central computer. Therefore, it is important that the data logger readings are recorded on the field sheet.

Acceptance criteria can be found in the Measurement Quality Acceptance Tables from the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Appendix D. EPA-454/B-13-003, May 2013: http://www.epa.gov/ttnamti1/files/ambient/pm25/qa/QA-Handbook-Vol-II.pdf.

2.14.4.2 Span Check (NO/NO_x)

At this point the zero air readings have been recorded, perform the span check. On the Main Screen (or Standby Screen) of the 146*i* Calibrator, press the **MENU** button to access the Main Menu Screen. Scroll to **OPERATION** and press **ENTER**. Use the ↓ button to scroll down to **SPAN**. Use the ← and → buttons to scroll to **SPAN 1** and press **ENTER**. **SPAN 1** has been programmed to produce the span level of 90% the upper range limit of the instrument (or 200 ppb NO/NO_x). Perform the span check using a cylinder containing 10-20 ±2 ppm NO in N_2 with less than 1 ppm NO_2. Begin turning the flow regulator on from the cylinder. Next, turn on the flow adjustment valve to produce sufficient flow. The flow can be read on the 146*i* Calibrator display screen by accessing the **DIAGNOSTICS FLOW** screen. This screen reports the actual gas and zero-air flows and the corresponding target flows.

Allow the 42*i* site analyzer to stabilize for a minimum of 15 minutes, then record the resultant data logger readings from the NO, NO_2, and NO_X channels on the Zero/Precision/Span Field Sheet.

Determine the analyzer drift by comparing the collected span concentration to the previously collected bi-weekly span concentration. Refer to Section 2.10.3 for acceptable control limits.

$$\text{Span Drift} = \frac{(\text{Current Analyzer Reading} - \text{Previous Analyzer Reading})}{\text{Previous Analyzer Reading}} \times 100$$

2.14.4.3 Precision Check (NO/NO_x)

At this point the span readings have been recorded, perform the precision level check. Use the ← and → buttons to scroll to **SPAN 5** and press **ENTER**. **SPAN 5** has been programmed to produce the precision level of approximately 20 ppb NO/NOx.

Allow the 42*i* site analyzer to stabilize for a minimum of 15 minutes, then record the resultant data logger readings from the NO, NO_2, and NO_X channels on the Verification Field Sheet.

Using the following equation to determine the percent difference between the data logger and the calibrator:

$$\%Difference = \frac{C_M - C_{146}}{C_{146}} \times 100$$

Where:

C_M = data logger concentration, ppb

C_{146} = Calibrator concentration, ppb

2.14.4.4 Zero/Precision/Span Checks (NO_2)

At this point the precision and span readings for NO/NO_x have been recorded, perform the Zero/Precision/Span Checks for NO_2. On the Main Screen (or Standby Screen) of the 146*i* Calibrator, press the **MENU** button to access the Main Menu Screen. Scroll to **OPERATION** and press **ENTER**. Use the ↓ button to scroll to the third line. Use the ← and → buttons to scroll to **OZON MAN**.

NOTE: Check and make sure that the second line is set to SPAN 1 which is programmed to obtain and NO_X concentration of 225 ppb. All NO_2 checks must be performed using the same settings as the span check for NO/NO_x.

Push the **MENU** button on the 146*i* Calibrator. Scroll to **OZONATOR** and press **ENTER**. Scroll to **MANUAL** and press **ENTER**. The ozone reading on the 146*i* Calibrator will be displayed as a percentage. Use the arrow keys to set the ozone level to 0%. Allow the 42*i* site analyzer to stabilize for a minimum of 15 minutes, then record the resultant data logger readings from the NO, NO_2, and NO_X channels on the Verification Field Sheet. NO_2 should stabilize around 0 ppb. NO/NO_X channels should stabilize around 225 ppb.

At this point, the zero NO_2 reading has been performed. Press the **MENU** button on the 146*i* Calibrator. Scroll to **OZONATOR** and press **ENTER**. Scroll to **MANUAL** and press **ENTER**. Use the arrow keys to adjust the ozonator percentage to obtain a NO_2 reading of approximately 200 ppb. Allow the 42*i* site analyzer to stabilize, then record the resultant data logger readings from the NO, NO_2, and NO_X channels on the Verification Field Sheet.

Use the arrow keys to adjust the ozonator percentage to obtain a NO_2 reading of approximately 20 ppb. Allow the 42*i* site analyzer to stabilize, then record the resultant data logger readings from the NO, NO_2, and NO_X channels on the Verification Field Sheet.

The actual NO_2 readings must be determined by using the formula:

$$NO_{2Actual} = (NO_{ori} - NO_{rem}) + \frac{F_{NO} * NO_{2imp}}{F_T}$$

Where:

NO_{ori} = Original NO concentration

NO_{rem} = NO conc. remaining after addition of O_3

F_{NO} = NO flow rate, sccm

NO_{2imp} = Conc. of NO_2 impurity in standard NO cylinder

F_T = Total flow required at output manifold, sccm

After determining the actual NO_2 readings for the precision check, calculate the percent difference using the formula:

$$\% \text{ Difference} = \frac{NO_{2\,(Indicated)} - NO_{2\,(Actual)}}{NO_{2\,(Actual)}} \times 100$$

Where:
$NO_{2\,(Indicated)}$ = data logger concentration, ppb

Turn off the gas from the cylinder standard.

Log all site visits into the logbook (every detail of the visit must be logged).

Refer to Section 2.14.2 to log out from the data logger.

2.14.4.5 Converter Efficiency Check

During each bi-weekly verification, the converter efficiency must be determined for the NO_2 precision and Span values. The converter efficiency slope criteria of 0.96 to 1.04 explicitly applies to each level. Use the Converter Efficiency Spreadsheet to determine if the verification passes, Form 7, Appendix A. See Appendix B for detailed instructions for using the Converter Efficiency Spreadsheet.

NOTE: If a problem is recognized while performing maintenance, diagnostic, zero air, weekly precision or span checks which has or could affect data, a corrective action form is to be filled out describing the problem identified and the action taken to correct the problem, Form 8, Appendix A. All forms and documentation must be submitted to the Quality Assurance Officer.

2.14.5 Investigate and Recalibrate Analyzer

If the precision check results in a percent difference $\pm$ 15%, or if the zero drift is $\geq$ 1.5 ppb, or if the span drift is $\pm$ 10%, the site analyzer must be recalibrated and the data will be invalidated back to the last valid zero/precision/span check. See Section 2.9 for calibration procedures. Record all calibration information in the log books.

NOTE: In order to minimize data loss, Polk County personal will recalibrate the instrument when the results of bi-weekly checks reach 80% of invalidation thresholds, unless there is compelling reason and justification not to do so.

APPENDIX A – Forms and Field Sheets

Form 1.....**Calibration Field Sheet**
Form 2.....**Calibration Linear Regression**
Form 3.....**Calibration Converter Efficiency**
Form 4.....**Audit Field Sheet**
Form 5.....**Audit Converter Efficiency**
Form 6.....**Verification Field Sheet**
Form 7.....**Verification Converter Efficiency**
Form 8.....**Corrective Action Form**

Form 1: Calibration Field Sheet

$NO-NO_2-NO_x$ Calibration Sheet

Site:_____

1)Routine Site Information

Date/Time:_____
Operator:_____

Analyzer Serial #:_____
Date of last calibration:_____
TS Serial # :_____
Date of last calibration:_____
Prec cylinder #:_____
Cylinder Expiration Date:_____
Zero Air Generator Serial #:_____
Date of last annual maintenance:_____
Cylinder > 200 psig **(Y or N)**
Cylinder Concentration:_____
Cylinder NO Concentration: _____
Cylinder NOx Concentration: _____
Cylinder impurities: _____
Station Observations Made **(Y or N)**
Changed Filter? **(Y or N)**
Moisture Trap Checked? **(Y or N)**
Sample Line Checked? **(Y or N)**
Data logger recording shelter temp? **(Y or N)**
Reading:_____

NO Reading (ppm):_____
NO2 Reading (ppm):_____
NOx Reading (ppm):_____
Date Last Calibration: _____
Ambient:_____

2) Diagnostic Check
ALARMS DETECTED:

Make adjustments per service manual

Alarm	Value	Corrective Action
Internal Temp (°C) (8 - 47°C)		Consult Manual
Chamber Temp (°C) (47 – 51 °C)		Check Reaction Chamber
Cooler Temp (°C) (-40 to 10°C)		Check PMT Cooler
Conv. Temp (°C) (300 - 700°C)		Check NO_2 to NO Converter
Pressure (150 - 300 mm Hg)		Replace Reaction Chamber
Sample Flow (0.350 – 0.900 LPM)		Replace Pump
Ozonator Flow (> 0.050 LPM)		Replace Pump
Leak Check (Flow = 0; Pressure < 250 mmHg)		Locate Leak Replace solenoid valves Replace Capillaries

Coefficients:

	NO BKG (< 15 ppb)	NOX BKG (15 < ppb)	NO COEF (0.900-1.100)	NO2 COEF (0.900-1.100)	NOx COEF (0.900-1.100)	PMT	Counts
Before							
After							

Note: Refer to Section 2.9 of Polk County Air Quality's Standard Operating Procedures $NO-NO_2-NO_x$ for exact calibration procedures.

Gas A Dilution – NO/NO_x

4) Zero Air Check

Target Total: _____sccm Actual Total: _____sccm
Zero Target: _____sccm Zero Actual: _____sccm
Gas Taraget:_____sccm Gas Actual: _____sccm

5) Point 1 NO/NOx (~200 ppb)
Transfer Std Actual: _____
Target Total: _____sccm Actual Total: _____sccm
Zero Target: _____sccm Zero Actual: _____sccm
Gas Taraget:_____sccm Gas Actual: _____sccm

6) Point 2 NO/NOx (~160 ppb)
Transfer Std Actual: _____
Target Total: _____sccm Actual Total: _____sccm
Zero Target: _____sccm Zero Actual: _____sccm
Gas Taraget:____ sccm Gas Actual: _____sccm

4)	NO	NO_2	NO_x	5)	NO	NO_2	NO_x	6)	NO	NO_2	NO_x
300 sec reading				300 sec reading				300 sec reading			
% diff.				% diff.				% diff.			

7) Point 3 NO/NOx (~120 ppb)
Transfer Std Actual:_____
Target Total: _____sccm Actual Total: _____sccm
Zero Target: _____sccm Zero Actual: _____sccm
Gas Taraget:_____sccm Gas Actual: _____sccm

8) Point 4 NO/NOx (~80 ppb)
Transfer Std Actual:_____
Target Total: _____sccm Actual Total: _____sccm
Zero Target: _____sccm Zero Actual: _____sccm
Gas Taraget:_____sccm Gas Actual: _____sccm

9) Point 5 NO/NOx (~40 ppb)
Transfer Std Actual: _____
Target Total: _____sccm Actual Total: _____sccm
Zero Target: _____sccm Zero Actual: _____sccm
Gas Taraget:_____sccm Gas Actual: _____sccm

7)	NO	NO_2	NO_x	8)	NO	NO_2	NO_x	9)	NO	NO_2	NO_x
300 sec reading				300 sec reading				300 sec reading			
% diff.				% diff.				% diff.			

GPT – NO_2

9)Zero Air Check
Transfer Standard : 0%

10) Point 1 (~200 ppb)
Transfer Std %:

11) Point 2 (~160 ppb)
Transfer Std %:

9)	NO	NO_2	NO_x	10)	NO	NO_2	NO_x	11)	NO	NO_2	NO_x
300 sec reading				300 sec reading				300 sec reading			
% diff.				% diff.				% diff.			

12) Point 3 (~120 ppb)
Transfer Standard % :

13) Point 4 (~80 ppb)
Transfer Standard %:

14) Point 5 (~40 ppb)
Transfer Standard %:

12)	NO	NO_2	NO_x	13)	NO	NO_2	NO_x	14)	NO	NO_2	NO_x
300 sec reading				300 sec reading				300 sec reading			
% diff.				% diff.				% diff.			

Form 2 – Calibration Linear Regression

NO/NOy-NO/NOx Multi-Point calibration spreadsheet

Analyzer:	**THERMO 42i**			
Serial No.:	**1207251984**		Operator	RP
Site:	**Carpenter**			
Full Scale:	**500**	**ppb**	Calibrator:	146i 1030945145
1% of Full Scale:	**5**	**ppb**	Cal Gas:	EA000737
Date:	**June 21, 2012**		Conc.	49.6 ppm
			Exp. Date	4/5/2013

NO Cal

Known Conc.	Measured Conc.	rpd	slope	intercept	rsq	forecast	distance to least squares line	acceptable distance	Pass/Fail
0	0	n/a	1.0108	0.0526	1.0000	0.0526	0.0526	5.000	pass
50.0	50.7	1.4%				50.5947	0.1053	5.000	pass
100	101	1.0%				101.1368	0.1368	5.000	pass
200	202	1.0%				202.2211	0.2211	5.000	pass
300	304	1.3%				303.30526	0.6947	5.000	pass
400	404	1.0%				404.38947	0.3895	5.000	pass

NOx Cal

Known Conc.	Measured Conc.	rpd	slope	intercept	rsq	forecast	distance to least squares line	acceptable distance	Pass/Fail
0	-0.2	n/a	1.0016	0.4274	1.0000	0.4274	0.6274	5.000	pass
50.0	50.4	0.8%				50.5053	0.1053	5.000	pass
100	101	1.0%				100.5832	0.4168	5.000	pass
200	202	1.0%				200.7389	1.2611	5.000	pass
300	300	0.0%				300.8947	0.8947	5.000	pass
400	401.0	0.3%				401.0505	0.0505	5.000	pass

NO2

Known Conc.	Measured Conc.	rpd	slope	intercept	rsq	forecast	distance to least squares line	acceptable distance	Pass/Fail
0	0	n/a	0.9994	0.6841	1.0000	0.6841	0.6841	5.000	pass
50.0	51	2.0%				50.6520	0.3480	5.000	pass
101.0	102	1.0%				101.6194	0.3806	5.000	pass
205.0	206	0.5%				205.5527	0.4473	5.000	pass
300	300	0.0%				300.4918	0.4918	5.000	pass
395.4	395.6	0.000505817				395.83065	0.2306	5.000	pass

Form 3 – Calibration Converter Efficiency

Cylinder Conc.	49.60	PPM
Impurity	0.10	PPM

ZERO TEST

	AIR	*GAS*	*Known Value*			*Instrument Response*		
Calibration	**Zero Air MFC**	**Pollutant MFC**	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
	SCCM	**SCCM**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
Zero	7993	0	0.0	0.0	0.0	0.3	-0.1	-0.2

NO, NOx UPSCALE CALIBRATION

	AIR	*GAS*	*Known Value*			*Instrument Response*		
Calibration	**Zero Air MFC**	**Pollutant MFC**	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
	SCCM	**SCCM**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
50 PPB	17985	17.92	49	49	-	49.7	49.6	-0.2
100 PPB	14876	29.84	99	99	-	101.0	100.6	-0.5
200 PPB	12566	49.64	195	196	-	201.9	200.8	-1.5
300 PPB	8051	48.66	298	299	-	302.5	301.9	-1.0
400 PPB	6237	50.45	398	399	-	401.4	400.9	-1.2

NO2 UPSCALE CALIBRATION (GPT)

Calibration	*Instrument Response*		
points NO2	**NO**	**NOx**	**NO2**
	PPB	**PPB**	**PPB**
Ozone Lamp Off (GPT Baseline)	450.9	452.9	1.1
50 PPB	400.6	453.3	52.3
100 PPB	355.0	453.2	98.2
200 PPB	253.1	452.9	199.8
300 PPB	155.6	454.0	298.8
400 PPB	54.5	453.3	398.9

CALIBRATION SUMMARY

	Known Values			*Instrument Response*		
Pollutant	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
Level	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
Zero	0.0	0.0	0.0	0.3	-0.1	-0.2
50 PPB	49.4	49.5	-	49.7	49.6	-0.2
100 PPB	99.3	99.5	-	101.0	100.6	-0.5
200 PPB	195.2	195.6	-	201.9	200.8	-1.5
300 PPB	298.0	298.6	-	302.5	301.9	-1.0
400 PPB	398.0	398.8	-	401.4	400.9	-1.2

NO2 CONVERTER EFFICIENCY TEST

Pollutant	**(ΔNO)corrected**	**(ΔNOx)corrected**	**(ΔNO2)corrected**	**Converter Efficiency (1pt)**	**Is Converter Efficiency 1 ±0.04 ?**
Level	**PPB**	**PPB**	**PPB**		
50 PPB	-49.8	0.4	50.2	100.8%	TRUE
100 PPB	-94.9	0.3	95.2	100.3%	TRUE
200 PPB	-195.7	0.0	19.7	100.0%	TRUE
300 PPB	-292.2	1.1	293.3	100.4%	TRUE
400 PPB	-392.2	0.4	392.6	100.1%	TRUE

Converter Efficiency (from slope)	**Is Converter Efficiency 1 ±0.04 ?**
100.10%	TRUE

Converter Efficiency OK ?
TRUE

Form 4 – Audit Field Sheet

$NO-NO_2-NO_x$ Audit Sheet

Site:_____

1)Routine Site Information

Date/Time:_____
Quarter/Year: _____
Operator/Auditor:_____
Analyzer Serial #:_____
Date of last calibration:_____
Audit TS Serial # :_____
Date of last calibration:_____
Prec cylinder #:_____
Cylinder Expiration Date:_____
Zero Air Generator Serial #:_____
Date of last annual maintenance:_____
Cylinder > 200 psig **(Y or N)**
Cylinder Concentration:_____
Cylinder NO Concentration: _____
Cylinder NOX Concentration: _____
Impurity in Tank: _____
Tank Expiration: _____
Station Observations Made? **(Y or N)**
Moisture Trap Checked? **(Y or N)**
Sample Line Checked? **(Y or N)**
Data logger recording shelter temp? **(Y or N)**
Reading:_____

Record NO BKG_____
Record NOx BKG:_____
Record NO Coef: _____
Record NO2 Coef:_____
Record NOx Coef:_____
Reading NO: _____
Reading NO2: _____
Reading NOx:_____
Date Last Zero/Prec/SpanCheck_____**Days**_____
Ambient:_____

2) Diagnostic Check
ALARMS DETECTED:

Make adjustments per service manual

Alarm	Value	Corrective Action
Internal Temp (°C) (8 - 47°C)		Consult Manual
Chamber Temp (°C) (47 – 51 °C)		Check Reaction Chamber
Cooler Temp (°C) (-40 to 10°C)		Check PMT Cooler
Conv. Temp (°C) (300 - 700°C)		Check NO_2 to NO Converter
Pressure (150 - 300 mm Hg)		Replace Reaction Chamber
Sample Flow (0.350 – 0.900 LPM)		Replace Pump
Ozonator Flow (> 0.050 LPM)		Replace Pump

Gas A Dilution – NO/NO_x

3)Zero Air Check
Transfer Std Actual:_____
Zero Flow Reading: _____sccms
Gas Flow Reading: sccms

4) Pt. 1 (3-5 ppb)
Transfer Std Actual: _____
Zero Flow Reading: _____sccm
Gas Flow Reading: sccm

5) Pt. 2 (6-100 ppb)
Transfer Std Actual: _____
Zero Flow Reading: _____sccm
Gas Flow Reading: sccm

6) Pt. 3 (110-250 ppb)
Transfer Std Actual: _____
Zero Flow Reading: _____sccm
Gas Flow Reading: sccm

3)	NO	NO_2	NO_x	4)	NO	NO_2	NO_x	5)	NO	NO_2	NO_x	6)	NO	NO_2	NO_x
300 sec reading				300 sec reading				300 sec reading				300 sec reading			
% diff				% diff				% diff				% diff			

If the percent difference is outside ±15% or if the absolute value of the zero reading is greater than 1.5 ppb, and there is reason to believe the results are incorrect or not representative of the analyzers performance, the cause for the error must be investigated and corrected. An investigation initiated by audit results outside the above limits may require the invalidation of data. Record all information in the site log.

$$\frac{DataLogger\ Averages\ (Y) - Calibrator\ (X)}{Calibrator\ (X)} \times 100 = \%_____\ Difference$$

Gas A Dilution – NO/NO_x

9)Zero Air Check
GPT %:_____
Zero Air Flow Reading: _____sccms
Gas Air Flow Reading: sccms

10) Pt. 1 (3-5 ppb)
GPT %: _____
Zero Air Flow Reading: _____sccm
Gas Air Flow Reading: sccm

11) Pt. 2 (6-100 ppb)
GPT %: _____
Zero Air Flow Reading: _____sccm
Gas Air Flow Reading: sccm

12) Pt. 3 (110-250 ppb)
GPT %: _____
Zero Air Flow Reading: _____sccm
Gas Air Flow Reading: sccm

9)	NO	NO_2	NO_x	10)	NO	NO_2	NO_x	11)	NO	NO_2	NO_x	12)	NO	NO_2	NO
300 sec reading				300 sec reading				300 sec reading				300 sec reading			
% diff				% diff				% diff				% diff			

If the percent difference is outside ±15% or if the absolute value of the zero reading is greater than 1.5 ppb, and there is reason to believe the results are incorrect or not representative of the analyzers performance, the cause for the error must be investigated and corrected. An investigation initiated by audit results outside the above limits may require the invalidation of data. Record all information in the site log.

$$NO_{2(Actual)} = (NO_{orig} - NO_{rem}) + NO_{2(imp)}$$

Where:
$NO_{2Actual}$ = Actual NO_2 concentration
$NO_{2(imp)}$ = Calculated NO impurities
NO_{orig} = Original NO datalogger reading, when NO_2 is set to zero
NO_{rem} = Remaining NO datalogger reading, after addition of O_3

$$NO_{2(imp)} = \frac{(F_{NO}\ (sccm))(\text{Impurities from cylinder gas (ppb)})}{F_O + F_D}$$

Where:
F_{NO} = NO Flow
F_O = Ozone Flow
F_D = Dilution Flow

Form 5 – Audit Converter Efficiency

Cylinder Conc.	49.60	PPM
Impurity	0.10	PPM

ZERO TEST

	AIR	*GAS*	*Known Value*			*Instrument Response*		
Audit	**Zero Air MFC**	**Pollutant MFC**	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
	SCCM	**SCCM**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
Zero	7993	0	0.0	0.0	0.0	0.0	-0.2	-0.2

NO, NOx UPSCALE Audit

	AIR	*GAS*	*Known Value*			*Instrument Response*		
Audit	**Zero Air MFC**	**Pollutant MFC**	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
	SCCM	**SCCM**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
2 - (30-80)	11047	19.74	88	89	-	46.4	44.9	-1.5
3 - (150-200)	7922	85.50	530	531	-	267.1	255.4	-12.1
4 - (350-450)	4912	84.20	836	838	-	422.5	401.6	-21.4

NO2 UPSCALE Audit (GPT)

Audit	*Instrument Response*		
points NO2	**NO**	**NOx**	**NO2**
	PPB	**PPB**	**PPB**
Ozone Lamp Off (GPT Baseline)	455.4	456.8	1.4
2 - (30-80)	400.0	456.2	55.4
3 - (150-200)	267.4	454.5	187.1
4 - (350-450)	66.2	453.7	387.5

Audit SUMMARY

	Known Values			*Instrument Response*		
Pollutant	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
Level	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
Zero	0.0	0.0	0.0	0.0	-0.2	-0.2
2 - (30-80)	88.5	88.7	-	46.4	44.9	-1.5
3 - (150-200)	529.6	530.7	-	267.1	255.4	-12.1
4 - (350-450)	835.9	837.6	-	422.5	401.6	-21.4

NO2 CONVERTER EFFICIENCY TEST

Pollutant	**(ΔNO) corrected**	**(ΔNOx) corrected**	**(ΔNO2) corrected**	**Converter Efficiency (1pt)**	**Is Converter Efficiency 1 ±0.04 ?**
Level	**PPB**	**PPB**	**PPB**		
2 - (30-80)	-109.9	-1.3	108.6	98.9%	TRUE
3 - (150-200)	-372.9	-4.8	368.0	98.7%	TRUE
4 - (350-450)	-771.9	-6.5	765.4	99.2%	TRUE

Converter Efficiency (from slope)	**Is Converter Efficiency 1 ±0.04 ?**
99.24%	TRUE

Converter Efficiency OK ?
TRUE

Form 6 – Verification Field Sheet

$NO-NO_2-NO_x$ Verification Sheet **Site:**_____

1)Routine Site Information

Date/Time:_____
Operator:_____

Analyzer Serial #:_____
Date of last calibration:_____
TS Serial # :_____
Date of last calibration:_____
Prec cylinder #:_____
Cylinder Expiration Date:_____
Zero Air Generator Serial #:_____
Date of last annual maintenance:_____
Cylinder > 200 psig **(Y or N)**
Cylinder Concentration:_____
Cylinder NO Concentration: _____
Cylinder NO_X Concentration: _____
Impurity in Tank: _____
Station Observations Made **(Y or N)**
Changed Filter? **(Y or N)**
Moisture Trap Checked? **(Y or N)**
Sample Line Checked? **(Y or N)**
Data logger recording shelter temp? **(Y or N)**
Reading:_____

Record NO BKG_____
Record NOx BKG:_____
Record NO Coef: _____
Record NO2 Coef:_____
Record NOx Coef:_____
Reading NO: _____
Reading NO2: _____
Reading NO_X: _____
Date Last Zero/Prec/SpanCheck_____**Days**_____
Ambient:_____

2) Diagnostic Check
ALARMS DETECTED:

Make adjustments per service manual

Alarm	Value	Corrective Action
Internal Temp (°C) (8 - 47°C)		Consult Manual
Chamber Temp (°C) (47 – 51 °C)		Check Reaction Chamber
Cooler Temp (°C) (-40 to 10°C)		Check PMT Cooler
Conv. Temp (°C) (300 - 700°C)		Check NO_2 to NO Converter
Pressure (150 - 300 mm Hg)		Replace Reaction Chamber
Sample Flow (0.6 – 0.8 LPM)		Replace Pump
Ozonator Flow (> 0.050 LPM)		Replace Pump

Gas A Dilution – NO/NO_x

4) Zero Air Check

Zero Flow Reading: _____sccms
Gas Flow Reading: _____sccms

4)	NO	NO_2	NO_x
300 sec reading			
Prev. Bi-weekly			
Drift			

If the difference in zero drift between the current indicated and previous bi-weekly indicated readings is outside ± 1.5 ppb, invalidate to last 0/span check and re-calibrate.

5) Precision Check (~20 ppb)

Transfer Std Actual:_____
Zero Flow Reading: _____sccm
Gas Flow Reading: _____sccm

5)	NO	NO_2	NO_x
300 sec reading			
% diff.			

If % difference between actual and indicated readings is outside ± 15%, invalidate to last 0/span check and re-calibrate.

6) Span Check (~200 ppb)

Transfer Std Actual: _____
Zero Flow Reading: _____sccm
Gas Flow Reading: _____sccm

6)	NO	NO_2	NO_x
300 sec reading			
Prev. Bi-weekly			
Drift			
% diff Drift			

If the span drift (defined in Section 2.14.4.2) is outside ± 10%, invalidate to last 0/span check and recalibrate.

$$\% \text{ Difference} = \frac{(\text{Current Data Logger Reading} - \text{Calibrator Reading})}{\text{Calibrator Reading}} \times 100$$

$$\text{Span Drift} = \frac{(\text{Current Analyzer Reading} - \text{Previous Analyzer Reading})}{\text{Previous Analyzer Reading}} \times 100$$

GPT – NO_2

9)Zero Air "Orig" Check Transfer Standard : 0%

10)Precision Check (~20 ppb) Transfer Std %:

11) Span Check (~200 ppb) Transfer Std %:

9)	NO	NO_2	NO_x	10)	NO	NO_2	NO_x	11)	NO	NO_2	NO_x
300 sec reading				300 sec reading				300 sec reading			
Previous bi-weekly								Previous bi-weekly			
Drift								Drift			
				Actual NO_2 :				Actual NO_2 :			
				% diff.				% diff. Drift			
If the difference in zero drift between the current indicated and previous bi-weekly indicated readings is outside ± 1.5 ppb, invalidate to last 0/span check and re-calibrate.				If % difference between actual and indicated readings is outside ± 15%, invalidate to last 0/span check and re-calibrate.				If the span drift (defined in Section 2.14.4.2) is outside ± 10%, invalidate to last 0/span check and recalibrate.			

$$NO_{2(Actual)} = (NO_{orig} - NO_{rem}) + NO_{2(imp)}$$

Where:
$NO_{2Actual}$ = Actual NO_2 concentration
$NO_{2(imp)}$ = Calculated NO impurities
NO_{orig} = Original NO conc, when NO2 is set to zero
NO_{rem} = Remaining NO conc, after addition of O_3

$$NO_{2(imp)} = \frac{(F_{NO}\,(sccm))(\text{Impurities from cylinder gas (ppb)})}{F_O + F_D}$$

Where:
F_{NO} = NO Flow
F_O = Ozone Flow
F_D = Dilution Flow

Note: Refer to Section 2.14 of Polk County Air Quality's Standard Operating Procedures NO-NO_2-NO_x for exact calibration procedures.

Form 7 – Verification Converter Efficiency

Cylinder Conc.	24.98	PPM
Impurity	0.00	PPM

ZERO TEST

	AIR	*GAS*	*Known Value*			*Instrument Response*		
Verification	**Zero MFC**	**Gas MFC**	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
	SCCM	**SCCM**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
Zero	8000	0	0.0	0.0	0.0	0.0	0.0	0.0

NO, NOx UPSCALE VERIFICATION

	AIR	*GAS*	*Known Value*			*Instrument Response*		
Verification	**Zero MFC**	**Gas MFC**	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
	SCCM	**SCCM**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
Precision	11957	43	90	90	-	89.0	88.6	-
Span (90% URL)	4910	90	450	450	-	454.7	454.6	-

NO2 UPSCALE VERIFICATION (GPT)

Verification	*Instrument Response*		
points NO2	**NO**	**NOx**	**NO2**
	PPB	**PPB**	**PPB**
Ozone Lamp Off (GPT Baseline)	453.5	454.4	-
Ozonator Mid	359.5	452.2	92.7
Ozonator High	54.1	450.3	396.2

VERIFICATION SUMMARY

	Known Values			*Instrument Response*		
Pollutant	**NO**	**NOx**	**NO2**	**NO**	**NOx**	**NO2**
Level	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**	**PPB**
Zero	0.0	0.0	0.0	0.0	0.0	0.0
Precision	90.0	90	94.0	89.0	88.6	92.7
Span	450.0	450	399.4	454.7	454.6	396.2

NO2 CONVERTER EFFICIENCY TEST

Pollutant	**(ΔNO) corrected**	**(ΔNOx) corrected**	**(ΔNO2) corrected**	**Converter Efficiency (1pt)**	**Is Converter Efficiency 1 ±0.04 ?**
Level	**PPB**	**PPB**	**PPB**		
Ozonator Mid	-92.9	-2.2	90.7	97.7%	TRUE
Ozonator High	-394.7	-4.1	390.6	99.0%	TRUE

Converter Efficiency (from slope)	**Is Converter Efficiency 1 ±0.04 ?**
99.38%	TRUE

Converter Efficiency OK ?
TRUE

Form 8 – Corrective Action Form

Polk County Air Quality - Corrective Action Form

To: _____ Polk County Air Quality _____
(position)

From: _____

Copies of completed form to: AQ Supervisor, File

Urgency: ☐ Emergency (immediate action needed) ☐ Urgent (24 Hr.)
☐ Routine (7 days) ☐ Next scheduled visit ☐ Information only

Problem Identification:

Site: _____
System: _____
Date: _____

Description of Problem: _____

Recommended Action: _____

Signature of Initiator: _____ Date: _____

Problem Resolution:

Date of Corrective Action: _____

Summary of Corrective Action: _____

Result of Corrective Action: _____

Signature of resolver: _____ Date: _____

Signature of QA Officer: _____ Date: _____

Appendix B: Instructions for Using the Converter Efficiency Check Spreadsheet

Note: The Converter Efficiency Check Spreadsheet, and following instructions, originated from the IDNR.

Converter Efficiency
During Gas phase titration, NO is converted to NO_2

$NO + O_3 \rightarrow NO_2 + O_2$

When the amount of ozone produced by the ozone lamp in the calibrator is increased, the NO_2 at the outlet goes up and the NO goes down by an equal amount (i.e. $\Delta NO_2 = -\Delta NO$, or $\Delta NO_2/|\Delta NO|=1$). If multiple changes in the ozonator are made then the slope of ΔNO_2 ***vs.*** $|\Delta NO|$ will be one for the same reason.

If an instrument is used to measure the NO and NO_2 at the outlet, the ratio of ΔNO_2 to $|\Delta NO|$ (or the slope of ΔNO_2 vs. $|\Delta NO|$) can be used to establish the converter efficiency, where ΔNO_2 is computed from the equation $\Delta NO_2 = \Delta NOx - \Delta NO$. If the instrument has not been recently calibrated, then the NO and NOx calibration curves will be used to establish the "true" instrument readings for ΔNOx and ΔNO in these equations.

Sequence in Field Procedure:
The spreadsheet assumes that a zero check will be done on all three channels first. Next, a precision check is done on the NO and NO_x channel, and then a span check is done on the NO and NO_x channel. The spreadsheet assumes that the mass flow controller settings used to obtain the span values for the NO and NO_x channels remain unchanged, until the completion of the gas phase titration. (cells D34 and D35 use those settings to calculate the effect of stock cylinder NO_2 impurity.)

Explanation of color highlighting:
- Pale Yellow – raw data entered by user
- White – results calculated by spreadsheet

User entered constants:

Complete fields on the tab labeled "Instrument Information". Note that the values entered for the NO concentration and NO_2 impurity in the stock cylinder need to be re-entered in the upper part of the tab labeled "Converter Efficiency".

User entered variables:

Zero Air MFC: Enter the flow through the zero air mass flow controller (MFC) of the Thermo 146i gas dilution calibrator in cubic centimeters per minute at EPA standard

temperature and pressure (SCCM). In the Thermo model 146*i*, this flow represents the sum of the dilution air and the flow past the ozone lamp.

Pollutant MFC: Enter the flow through the pollutant mass flow controller of the gas dilution calibrator in SCCM.

Instrument Response (NO ppb, NO_x ppb, and NO_2 ppb): Although the spreadsheet uses the header "Instrument Response" to designate indicated values, these measurements are normally taken from the data logger rather than the analyzer.

Fields calculated by spreadsheet:

Known Value (NO ppb): calculated as cylinder NO concentration * [Pollutant MFC/(Pollutant MFC + Zero Air MFC)] flows. Since the cylinder NO concentration is given in ppm, multiply the product by 1,000 to convert from ppm to ppb. The known NO values from the "ZERO TEST" and "NO, NOx UPSCALE VERIFICATION" tables are copied to the "VERIFICATION SUMMARY" table.

Known Value (NO_x ppb): calculated as (sum of cylinder NO concentration and cylinder NO2 impurity) * [Pollutant MFC/(Pollutant MFC + Zero Air MFC)] flows. Since the cylinder NO_x concentration is given in ppm, multiply the product by 1,000 to convert from ppm to ppb. The known NO_x values from the "ZERO TEST" and "NO, NOx UPSCALE VERIFICATION" tables are copied to the "VERIFICATION SUMMARY" table.

Known Value (NO_2 ppb): The known NO_2 value from the "ZERO TEST" table is copied to the "VERIFICATION SUMMARY" table. At the NO_2 precision level, the known NO_2 value is calculated as (NO when the ozone lamp is off) – (NO with O_3 lamp at mid-level setting), plus the diluted effect of any NO_2 impurity in the stock cylinder. At the NO_2 span level, the known NO_2 value is calculated as (NO when the ozone lamp is off) – (NO with O_3 lamp at high-level setting), plus the diluted effect of any NO_2 impurity in the stock cylinder. The effect of NO_2 impurity in the stock cylinder is calculated as cylinder NO_2 concentration * [Pollutant MFC/(Pollutant MFC + Zero Air MFC)] flows. Since the cylinder NO_2 concentration is given in ppm, multiply the product by 1,000 to convert from ppm to ppb. MFC flow settings during the NO_2 precision and span point are assumed to be the same as those used for the NO and NO_x span check.

(ΔNO)corrected ppb: These cells only appear in the "NO_2 CONVERTER EFFICIENCY TEST" table. The delta, or Δ symbol, represents a difference defined as a final value minus initial value. The formula in the cells under the ***(ΔNO)corrected*** heading accomplishes two things. First, the instrument response for NO is corrected based on the known values for NO. Ordinarily this would be done by using the slope (m) and intercept (b) from a plot with the known values on the X axis and the instrument response on the Y axis. Secondly, the drop in NO between the baseline level (O_3 lamp off) and the "lamp on" settings is calculated. Because the correction equation is based on a straight line, the Y intercept drops out:

$$[(NO_{final} - b)/m] - [(NO_{initial} - b)/m] = (NO_{final} - NO_{initial})/m = (\Delta NO)corrected$$

(ΔNOX)corrected ppb: These cells only appear in the "NO_2 CONVERTER EFFICIENCY TEST" table. The formula in the cells under the ***(ΔNOX)corrected*** heading accomplishes two things. First, the instrument response for NO_X is corrected based on the known values for NO_X. Ordinarily this would be done by using the slope (m) and intercept (b) from a plot with the known values on the X axis and the instrument response on the Y axis. Secondly, the drop in NO_X between the baseline level (O_3 lamp off) and the "lamp on" settings is calculated. Because the correction equation is based on a straight line, the Y intercept drops out:

$$[(NO_{Xfinal} - b)/m] - [(NO_{Xinitial} - b)/m] = (NO_{Xfinal} - NO_{Xinitial})/m = (\Delta NOX)corrected$$

(ΔNO2)corrected ppb: These cells only appear in the "NO2 CONVERTER EFFICIENCY TEST" table. The formulas in the cells under the ***(ΔNO2)corrected*** heading subtract ***(ΔNO)corrected*** from ***(ΔNOX)corrected.*** As a result of the increase in ***NO_2*** from turning on the ozone lamp, the sign of the difference is positive. Note that the actual increase in the NO_2 concentration, that results from turning on the ozone lamp, will be greater than ***(ΔNO2)corrected*** if the converter is inefficient. ***(ΔNO2)corrected*** only reflects the increase in NO_2 measured by the analyzer.

Final Results and Acceptance Criteria:

Converter Efficiency (1pt): Cells in this column compute the ratio of ***(ΔNO2)corrected*** *to the absolute value of* ***(ΔNO)corrected.***

Is Converter Efficiency 1 ±0.04 ?: The acceptable range for Converter Efficiency (CE) is 96% to 104%. Values that round up to 96%, or down to 104%, are also acceptable. The cell evaluates to true if this acceptance criteria for converter efficiency is met.

Converter Efficiency (from slope): Section 1.5.10.1 of Appendix F, Part 50, 40CFR defines the converter efficiency as a slope. This cell calculates and displays the overall converter efficiency as the slope of a plot generated using ***(ΔNO2)corrected*** as the Y axis, and ***(ΔNO)corrected*** as the X axis. Note that this value is typically not the same as the mathematical average of the 1-point values for converter efficiency.

Converter Efficiency OK ?: The converter efficiency test passes only if both of the 1-point converter efficiencies (corresponding to the precision and span level), and the converter efficiency from the slope evaluate to TRUE.