

Standard Operation Procedures for NO-NO₂-NO_x

In Use By

**Polk County Air Quality
Ambient Air Monitoring Personnel**

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POLK COUNTY AIR QUALITY DIVISION

STANDARD OPERATING PROCEDURE MANUAL FOR THERMO SCIENTIFIC NO-NO₂-NO_x MONITORING

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2.0 STANDARD OPERATING PROCEDURE FOR NO-NO₂-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-NO₂-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, bi-weekly zero/precision/span checks, calibrations, audits and maintenance of Thermo Fisher Scientific's Trace Level 42i analyzers equipment operated by Polk County Air Quality personnel.

2.3 References

- 2.3.1 Thermo Scientific, Model 42i Trace Level, Chemiluminescence NO-NO₂-NO_x Analyzer, Instruction Manual. Part Number 102855-00, December 20, 2007.**
- 2.3.2 Thermo Electron Corporation, Model 146i, 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 EPA-454/B-13-003, July 2014. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II. Ambient Air Quality Monitoring Program, Appendix D.**
- 2.3.8 40 Code of Federal Regulations (CFR) Part 50, Appendix F, Measurement Principle and Calibration Procedure for the Measurement of NO_x in the Atmosphere.**
- 2.3.9 40 Code of Federal Regulations (CFR) Part 58, Appendix A, Quality Assurance Requirements for State and Local Air Monitoring Stations (Slams).**
- 2.3.10 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 Assessment Monitoring Stations (PAMS).**
- 2.3.11 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 (NO₂) is a health and environmental concern. For this reason, National Ambient Air Quality Standards (NAAQS) have been

established for NO₂. As a result, an air monitoring network to monitor for NO₂ was established in the Des Moines Metropolitan Statistical Area (MSA) and is maintained by Polk County Air Quality Division, Iowa.

Measurements of nitrogen dioxide (NO₂) in ambient air are based on the principle that nitric oxide (NO) and ozone (O₃) react to produce a characteristic luminescence with an intensity linearly proportional to the NO concentration. Infrared light emission results when electronically excited NO₂ molecules decay to lower energy states:



Ambient air is drawn into the NO-NO₂-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 NO₂ 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 O₃. The O₃ reacts with the NO present in each sample producing electronically excited NO₂ molecules. A photomultiplier tube housed in a thermoelectric cooler detects the NO₂ 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 NO₂ concentration calculated by difference. (NO_x – NO = NO₂).

The dynamic parameter requirement ensures that the NO-O₃ 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 146i Instruction Manual.

2.5 Health and Safety Warning/Precautions

Only properly trained personnel should perform TL-42i 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 TL-42i. 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 NO₂, it is important to understand that the reactive oxides of nitrogen in the atmosphere are primarily nitric oxide (NO) and nitrogen dioxide (NO₂), known together

as NO_x. During the daytime, there is a rapid interconversion of NO and NO₂. NO emitted into the air is converted to NO₂ 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 NO₂.

For more detailed information concerning site selection for NO-NO₂-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-NO₂-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.
- 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 NO₂ 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 NO₂ 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-NO₂-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 Scientific Model 42i Trace Level (TL-42i) Chemiluminescence NO-NO₂-NO_x Analyzer displayed in Figure 3-1 for sampling nitrogen dioxide. Installation of the TL-42i consists of connecting the sample tubing to the sample gas inlet fitting and connecting the primary power and the data logger device. An external pump must also be plumbed to the instrument. The sampler inlet line connection should be made with ¼ -inch outer diameter Teflon tubing.

Figure 2-1: Thermo Scientific TL-42i NO-NO₂-NO_x Analyzer



The Thermo TL-42i runs on an external twin-head vacuum pump. Connect the pump vacuum port (inlet) to the Exhaust bulkhead. Connect the pump exhaust to a suitable vent or charcoal scrubber.

Because the analyzer is an optical instrument, it is possible that particulate in the gas sample could interfere with the NO-NO₂-NO_x readings, even though 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, a Teflon filter must be installed between the ambient sample line and the sample port of the analyzer prior to operation. A 0.5-micron Teflon filter will not degrade the NO₂ concentration. However, if particulate matter builds up on the filter, the particulate matter will destroy some of the NO₂ in the sample.

Since the instrument's exhaust consists of ambient air with some NO₂ 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 TL-42i

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-NO₂-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 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", and press ENTER. Press the ↓ button until the cursor is on SET CUSTOM RANGES, and press ENTER. Press the ↓ button to scroll to CUSTOM RANGE 1 and press ENTER. Use the ← and → buttons to move the cursor left or right. Use the ↑ or ↓ buttons to increment or decrement the numeric value to set the range to "0.250" and press ENTER. Press MENU to return to the Range Menu. Repeat procedures to set NO₂ Range and NO_x Range.

The Gas Units Screen defines how the NO-NO₂-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-NO₂-NO_x measurements are taken. The average concentration of the readings is calculated for that time period. Polk County Air Quality will use 300 seconds as the averaging time for the collection of data. An averaging time of 60 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. Polk County Air Quality will run the NO-NO₂-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-NO₂-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 compensation 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.

3.6.4.7 Auto Mode

The Auto/Manual Mode screen allows selection of the automatic mode (NO/NO_x), NO Mode (manual NO), or NO_x mode (manual NO_x). **Polk County Air Quality will always run the TL-42i in Auto Mode.** The auto cycle mode switches the mode solenoid valves automatically on a 10 second cycle so that NO, NO₂, and NO_x concentrations are determined.

To set the TL-42i to Auto Mode, 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 **AUTO/MANUAL MODE** and press **ENTER**. Use the ↓ buttons to scroll to **NO/NO_x MODE** and press **ENTER**.

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

2.7 Standards

A cylinder containing 10 to 20 ppm NO in N₂ with less than 1.0 ppm NO₂ is used as the concentration standard for verifications, calibrations and audits. Cylinders must be traceable to a National Institute of Standards and Technology (NIST) NO in N₂ Standard Reference Material or NO₂ Standard Reference Material. Procedures for certifying the NO cylinder (working standard) against a NIST traceable NO or NO₂ standard and for determining the amount of NO₂ impurity are given in EPA Publication NO. EPA-006/4-75-003, "Technical Assistance Document for the Chemiluminescence Measurement of Nitrogen Dioxide." 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. 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 146i with the new stock gas concentration. Adjust the 146i 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. Record all information on the Gas Comparison Spreadsheet, Form 1, Appendix A.

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

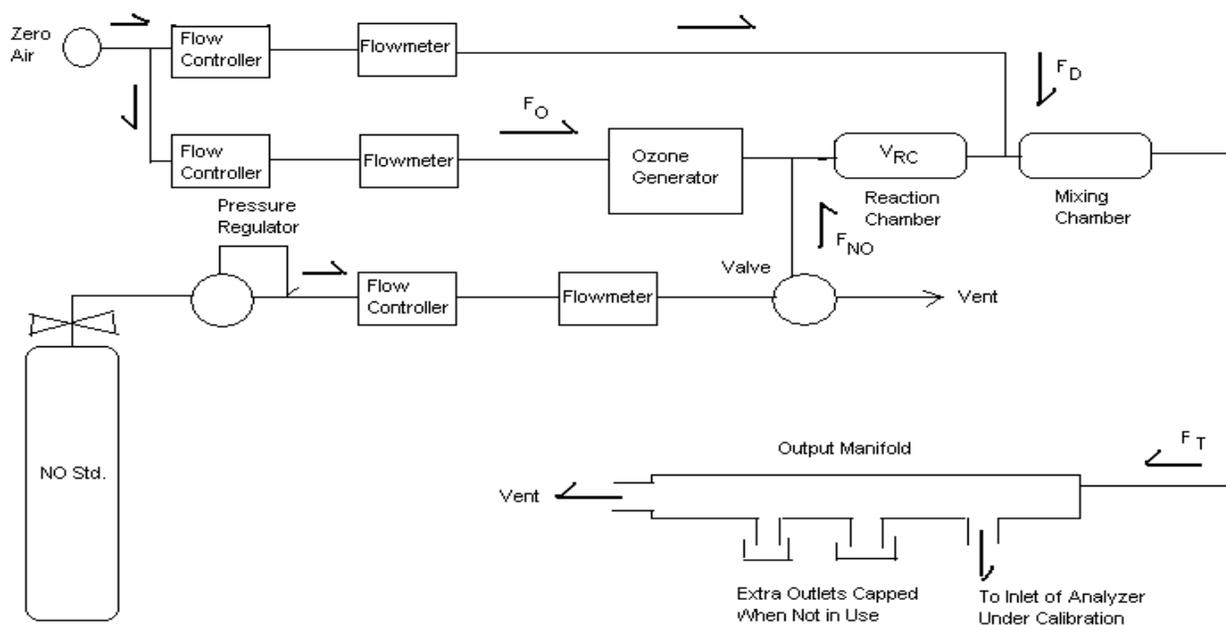
converting to NO₂ within the regulator resulting in errors during calibration. Better results will be achieved by alternately pressurizing and depressurizing the regulator once it has been attached to the cylinder.

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.

Figure 2-2: Gas Phase Titration System



2.8 Calibrations

The Thermo Scientific Model TL-42i NO-NO₂-NO_x monitor is calibrated whenever the following occurs: installation of a new monitor, instrument repair, when a zero/precision/span check fail to meet acceptance criteria limits, after 6 months has expired since the last calibration, or more frequently at the field operator’s discretion.

The Purpose of the calibration is to determine the NO, NO₂ and NO_x relationship between the analyzer and the true NO and NO₂ concentrations. This procedure involves the gas phase titration (GPT) of a NO standard with O₃ to produce NO₂. This method is based upon the rapid gas phase reaction between NO and O₃ to produce stoichiometric quantities of NO₂.



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-NO₂-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.

2.8.1 Calibration Procedures for the TL-42*i* using the 146*i* Calibrator

2.8.1.1 Pre-Calibration

Change the averaging time to 60 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 60 seconds. Press **RUN** to return to the Main Screen.

Make sure that the ozonator in the TL-42*i* is on. Press the front panel **MENU** button on the TL-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.

2.8.1.2 Calibration of NO/NO_x

The GPT requires the use of the NO/NO_x channel of the analyzer to determine the amount of NO₂ generated by titration. Therefore, it is necessary to calibrate and determine the linearity of the NO/NO_x channels before proceeding with the NO₂ 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, NO₂, and NO_x channels.

Press the front panel **MENU** button on the TL-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 PREREACTION ZERO** and press **ENTER**. Press **ENTER** to set the prereactor reading to zero. Press the **MENU** button to return to 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, NO₂, and NO_x channels.

Press the front panel **MENU** button on the TL-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, NO_x, and Prereactor Background coefficients should be less than 15 ppb. The NO and NO_x span coefficients should always fall between 0.900 and 1.100, and the NO₂ span coefficient should be between 0.960 and 1.100. If outside that range, refer to Thermo Scientific's Instruction Manual Chapter 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, NO₂, and NO_x channels.

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

2.8.1.3 Calibration of NO₂

On the main screen of the 146i 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 NO₂ calibration must be performed using an NO_x concentration of 225 ppb.

Push the **MENU** button on the 146i Calibrator. Scroll to **OZONATOR SETUP** and press **ENTER**. Scroll to **MANUAL** and press **ENTER**. The ozone reading on the 146i 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, NO₂, and NO_x channels. NO₂ 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 NO₂ 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 NO₂ reading in the range of 200 ppb. Allow the site analyzer to stabilize for a minimum of 15 minutes and record all data logger readings from the NO, NO₂, and NO_x channels.

NO₂ is calibrated according to a direct comparison between the NO concentration obtained when NO₂ is set to zero and the NO concentration obtained at approximately 80% the URL of the uncalibrated NO₂ channel (i.e. 200).

$$\text{NO}_{2\text{Actual}} = (\text{NO}_{\text{orig}} - \text{NO}_{\text{rem}}) + \text{NO}_{2(\text{imp})}$$

Where:

NO_{orig} = Original NO data logger reading, when NO₂ is set to zero

NO_{rem} = Final NO data logger reading when NO₂ is approx. 200 ppb

NO_{2(imp)} = Conc. of NO₂ impurity in standard NO cylinder

NOTE: To ensure that NO is in excess during Gas Phase Titration, NO_{rem} should be at least 10% of the value of NO_{orig}.

The NO₂ impurities in the NO concentration standard are determined by:

$$\text{NO}_{2(\text{imp})} = \frac{(\text{F}_{\text{NO}} (\text{sccm}))(\text{Impurities from cylinder gas (ppb)})}{\text{F}_{\text{NO}} + \text{F}_{\text{O}} + \text{F}_{\text{D}}}$$

Where:

F_{NO} = NO Flow

F_O = Ozone Flow

F_D = Dilution Flow

NOTE: The flow through the zero air mass flow controller of the Thermo 146i calibrator represents the sum of the dilution air and the flow past the ozone lamp.

Press the front panel **MENU** button on the TL-42i site analyzer to display the Main Menu. Use the arrow keys to select **CALIBRATION** and press **ENTER**. Scroll to **CALIBRATE NO₂ 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 NO₂ concentration. Press **ENTER** to calibrate the analyzer to the desired NO₂ concentration.

NOTE: If the analyzer calculates a NO₂ span coefficient of less than 0.96, either the entered NO₂ 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 2, 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 3, Appendix A.

Record all information in the site log.

2.8.2 Converter Efficiency (NO₂)

During a calibration, the converter efficiency must be determined for each NO₂ level. However, the critical criteria of 0.96 to 1.04 ultimately applies to the slope of the calibration curve. It is the average converter efficiency from the calibration curve which is used to determine if the converter needs serviced or replaced. Use the Converter Efficiency Spreadsheet to determine if the calibration passes, Form 4, Appendix A.

For more detailed information concerning calibration of a Thermo Scientific TL-42i NO-NO₂-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 42i, Chemiluminescence NO-NO₂-NO_x Analyzer.

2.9 Quality Control

2.9.1 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-NO₂-NO_x analyzer.

2.9.2 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.

2.9.3 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 146i 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 $\pm 15\%$, or if the zero drift is ≥ 5.0 ppb, or if the span drift is $\pm 10\%$. Acceptance criteria can be found in Table 2-2: The Measurement Quality Acceptance Tables from the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Appendix D. EPA-454/B-13-003, July 2014.

These criteria and corrective actions apply to both the manual and nightly auto-checks. For assessing bi-weekly zero and span drift, the current measured instrument response will be compared to the current known value. Corrective action will be taken if the difference is outside of historically established control limits.

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 2.13.4.

2.9.4 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 connect to the monitor site and examine the data recorded at the monitoring station (i.e. NO-NO₂-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 NO₂ standard, data averages of no longer than 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.

2.10 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 TL-42i and the 146i Calibrator, Teledyne 701H Zero Air Generator, and the Agilaire Model 8832 data logger. A flow controller with a flow rate regulated \pm 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.

2.10.1 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.

2.10.1.1 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.

2.10.1.2 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.

2.10.1.3 Particulate Filter Changes

A 0.5 micron Teflon filter housed in a Teflon filter holder is located on the outside of the analyzer and 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.

2.10.1.4 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.

2.10.1.5 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.

2.10.1.6 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-NO₂-NO_x concentrations due to contamination in the line, or every two years.

2.10.2 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 TL-42i, Chemiluminescence NO-NO₂-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.

2.10.3 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 20-30°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 20-30°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 (20-30°C). Data collected during temperatures outside of this range may be subject to invalidation, in accordance with the May 2013 Redbook's guidelines for operational criteria.

2.11 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-NO₂-NO_x analyzer once per quarter.

2.11.1 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)," will audit the NO-NO₂-NO_x analyzer 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 than 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 TL-42i continuous analyzer with known concentrations of NO-NO₂-NO_x within the measurement range of the analyzer. The 146i Multi-Gas Calibrators can be programmed to generate the desired NO-NO₂-NO_x audit concentrations, see Polk County Air Quality SOP Section 26.

Generate at least three of the following CFR 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, NO₂, and NO_x channels. The results are recorded on the Quarterly Audit Sheet, Form 5, Appendix A. The NO₂ Converter Efficiency should also be determined during an audit. Use the Converter Efficiency Spreadsheet to determine if it passes, Form 6, Appendix A. During each audit, the converter efficiency must be determined for each audit level. However, the critical criteria of 0.96 to 1.04 ultimately applies to the slope of the curve. It is the average converter efficiency from the curve which is used to determine if the converter needs serviced or replaced.

The audit fails if any of the following acceptance criteria are not met:

- The percent difference is outside ±15% for EPA Memo levels 3-10, and CFR levels of an equivalent concentration range.

- For EPA memo levels 1 and 2, and CFR levels of an equivalent concentration range; the acceptance criteria is ± 1.5 ppb, or $\pm 15\%$, whichever is less stringent.

If there is no reason to believe the results are incorrect or not representative of the analyzers performance, the cause for the audit failure must be investigated and corrected. In addition, if the absolute value of the zero reading is greater than 1.5 ppb during an audit, the results must also be investigated. 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-NO₂-NO_x monitoring, refer to the “Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II” or 40 CFR 58.

2.11.2 Performance Evaluation Audit

Agency staff may be sent an audit device, such as for the National Performance Evaluation Program (NPEP). The agency staff does not know the NO-NO₂-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-NO₂-NO_x and in all other EPA audit programs, which may arise in the future. According to the May 2013 Redbook, The audit fails if any of the following acceptance criteria are not met:

- The percent difference is outside $\pm 15\%$ for EPA Memo levels 3-10, and CFR levels of an equivalent concentration range.
- For EPA memo levels 1 and 2, and CFR levels of an equivalent concentration range; the acceptance criteria is ± 1.5 ppb, or $\pm 15\%$, whichever is less stringent.

If there is no reason to believe the results are incorrect or not representative of the analyzers performance, the cause for the audit failure 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.

2.12 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).

2.12.1 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 2.13). Calculations to assess precision are given below and should be used to assess precision on a quarterly basis. It is recommended that the NO-NO₂-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)".

2.12.2 Accuracy

The Polk County Air Quality Program participates in the USEPA performance audit program for NO₂ 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.

2.12.3 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.

2.13 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 42i Trace Level, Chemiluminescence NO-NO₂-NO_x Analyzer, Instruction Manual, and Model 146i, Dynamic Gas Calibration System, Instruction Manual.

2.13.1 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.

2.13.2 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



2.13.3 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 TL-42i 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, NO₂, 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 NO₂ 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

2.13.4 Bi-Weekly Zero, Precision and Span Checks

2.13.4.1 Zero Air Check

The zero air must be free of contaminants that could cause a detectable response on the TL-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 TL-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, NO₂, and NO_x channels on the Verification Field Sheet, Form 7, 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.

Determine the analyzer zero drift by comparing the collected zero concentration to the actual Calibrator concentration. Refer to Section 2.9.3 for acceptable control limits.

$$\text{Zero Drift} = C_M - C_{146}$$

Where:

C_M = data logger concentration, ppb

C_{146} = Calibrator concentration, ppb

2.13.4.2 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 146i 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 225 ppb NO/NO_x). Perform the span check using a cylinder containing 10-20 ±2 ppm NO in N₂ with less than 1 ppm NO₂. 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 146i 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 TL-42i site analyzer to stabilize for a minimum of 15 minutes, then record the resultant data logger readings from the NO, NO₂, and NO_x channels on the Zero/Precision/Span Field Sheet.

Determine the analyzer span drift by comparing the collected span concentration to the actual Calibrator concentration. Refer to Section 2.9.3 for acceptable control limits.

$$\text{Span Drift} = \frac{C_M - C_{146}}{C_{146}} \times 100$$

Where:

C_M = data logger concentration, ppb

C_{146} = Calibrator concentration, ppb

2.13.4.3 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/NO_x.

Allow the TL-42i site analyzer to stabilize for a minimum of 15 minutes, then record the resultant data logger readings from the NO, NO₂, 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:

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

Where:

C_M = data logger concentration, ppb

C_{146} = Calibrator concentration, ppb

Acceptance criteria can be found in Table 2-2: The Measurement Quality Acceptance Tables from the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Appendix D. EPA-454/B-13-003, July 2014.

2.13.4.4 Zero/Precision/Span Checks (NO₂)

At this point the precision and span readings for NO/NO_x have been recorded, perform the Zero/Precision/Span Checks for NO₂. On the Main Screen (or Standby Screen) of the 146i 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 NO₂ checks must be performed using the same settings as the span check for NO/NO_x.

Push the **MENU** button on the 146i Calibrator. Scroll to **OZONATOR** and press **ENTER**. Scroll to **MANUAL** and press **ENTER**. The ozone reading on the 146i Calibrator will be displayed as a percentage. Use the arrow keys to set the ozone level to 0%. Allow the TL-42i site analyzer to stabilize for a minimum of 15 minutes, then record the resultant data logger readings from the NO, NO₂, and NO_x channels on the Verification Field Sheet. NO₂ should stabilize around 0 ppb. NO/NO_x channels should stabilize around 225 ppb.

At this point, the zero NO₂ reading has been performed. Press the **MENU** button on the 146i 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 NO₂ reading of approximately 200 ppb. Allow the TL-42i site analyzer to stabilize, then record the resultant data logger readings from the NO, NO₂, and NO_x channels on the Zero/Precision/Span Field Sheet.

Use the arrow keys to adjust the ozonator percentage to obtain a NO₂ reading of approximately 20 ppb. Allow the TL-42i site analyzer to stabilize, then record the resultant data logger readings from the NO, NO₂, and NO_x channels on the Verification Field Sheet.

Using the NO Verification relationship determined in Section 2.13.4.1 - 2.13.4.3, correct NO_{ORIG} and NO_{REM} for slope and intercept prior to calculating NO_{2(ACTUAL)}. The NO_{2(ACTUAL)} readings are then determined by using the formula:

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

Where:

NO_{orig} = Original NO data logger reading

NO_{rem} = NO data logger reading, after addition of O₃

F_{NO} = NO flow rate (sccm) as determined by the pollutant MFC

NO_{2imp} = Conc. of NO₂ impurity in standard NO cylinder

F_T = Total actual flow put out by the calibrator, sccm

After determining the actual NO₂ 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:

NO₂ (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 2.13.2 to log out from the data logger.

2.13.4.5 Converter Efficiency Check

During each bi-weekly verification, the converter efficiency must be determined for the NO₂ precision and Span values. However, the critical criteria of 0.96 to 1.04 ultimately applies to the slope of the verification curve. It is the average converter efficiency from the verification curve which is used to determine if the converter needs serviced or replaced. Use the Converter Efficiency Spreadsheet to determine if the verification passes, Form 8, Appendix A.

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 9, Appendix A. All forms and documentation must be submitted to the Quality Assurance Officer.

2.13.5 Investigate and Recalibrate Analyzer

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 $\pm 15\%$, or if the zero drift is ≥ 5.0 ppb, or if the span drift is $\pm 10\%$. See Section 2.8 for calibration procedures.

NOTE: In order to minimize data loss, Polk County personal will recalibrate the instrument when the results of bi-weekly checks reach recalibration thresholds, unless there is compelling reason and justification not to do so. Recalibration criteria refer to thresholds that typically require recalibrating the analyzer, but do not require the invalidation of data. Recalibration criteria should be set to levels that are as tight as practically possible, and based on historical performance data. They may change after reviewing typical differences from control charts.

Table 2-2: Measurement Quality Objectives

NO₂, NO_x, NO Validation Template

1) Requirement (NO ₂)	2) Frequency	3) Acceptance Criteria	Information /Action
CRITICAL CRITERIA- NO₂			
<i>One Point QC Check Single analyzer</i>	1/2 weeks	≤ ±15% (percent difference)	1 and 2) 40 CFR Part 58 App A Sec 3.2 3) Recommendation based on DQO in 40 CFR Part 58 App A Sec 2.3.1.5 QC Check Conc range 0.01 - 0.10 ppm Relative to routine concentrations
Zero/span check	1/2 weeks	Zero drift ≤ ± 3.0 ppb (24 hr) ≤ ± 5.0 ppb (>24hr-14 day) Span drift ≤ ± 10 %	1 and 2) QA Handbook Volume 2 Section 12.3 3) Recommendation and related to DQO
<i>Converter Efficiency</i>	During multi-point calibrations, span and audit 1/2 weeks	(≥96%) 96% - 104%	1) 40 CFR Part 50 App F Section 1.5.10 and 2.4.10 2) Recommendation 3) 40 CFR Part 50 App F Section 1.5.10 and 2.4.10 Regulation states ≥96%, 96 - 104% is a recommendation.
OPERATIONAL CRITERIA- NO₂			
Shelter Temperature Range	Daily (hourly values)	20 to 30° C. (Hourly avg) or per manufacturers specifications if designated to a wider temperature range	1, 2 and 3) QA Handbook Volume 2 Section 7.2.2 Generally the 20-30 ° C range will apply but the most restrictive operable range of the instruments in the shelter may also be used as guidance. FRM/FEM list found on AMTIC provides temp. range for given instrument. FRM/FEM monitor testing is required at 20-30 ° C range per 40 CFR Part 53.32
Shelter Temperature Control	Daily (hourly values)	≤ ± 2° C SD over 24 hours	1, 2 and 3) QA Handbook Volume 2 Section 7.2.2
Shelter Temperature Device Check	1/6 mo	± 2° C of standard	1, 2 and 3) QA Handbook Volume 2 Section 7.2.2
<i>Annual Performance Evaluation Single Analyzer</i>	Every site 1/year 25 % of sites quarterly	Percent difference of audit levels 3-10 ≤ ±15% Audit levels 1&2 ± 1.5 ppb difference or ±15%	1) 40 CFR Part 58 App A sec 3.2.2 2) 40 CFR Part 58 App A sec 3.2.2 3) Recommendation - 3-audit concentrations not including zero. AMTIC guidance 2/17/2011 http://www.epa.gov/ttn/amtic/cpreldoc.html
<i>Federal Audits (NPAP)</i>	1/year at selected sites 20% of sites audited	Audit levels 1&2 ± 1.5 ppb difference all other levels percent difference ± 15%	1) 40 CFR Part 58 App A sec 2.4 2) NPAP adequacy requirements on AMTIC 3) NPAP QAPP/SOP
<i>Verification/Calibration</i>	Upon receipt/adjustment/repair/ installation/moving 1/6 months if manual zero/span performed biweekly 1/year if continuous zero/span performed	Instrument residence time ≤ 2 min Dynamic parameter ≥ 2.75 ppm-min All points within ±2 % of calibration range of best-fit straight line	1) 40 CFR Part 50 App F 2 and 3) Recommendation Multi-point calibration (0 and 4 upscale points)

1) Requirement (NO ₂)	2) Frequency	3) Acceptance Criteria	Information /Action
	daily		
<i>Gaseous Standards</i>	All gas cylinders	NIST Traceable (e.g., EPA Protocol Gas) 50-100 ppm of NO in Nitrogen with < 1 ppm NO ₂	1) 40 CFR Part 50 App F Section 1.3.1 2) NA Green book 3) 40 CFR Part 50 App F Section 1.3.1 Gas producer used must participate in EPA Ambient Air Protocol Gas Verification Program 40 CFR Part 58 App A sec 2.6.1
<i>Zero Air/ Zero Air Check</i>	1/year	Concentrations below LDL	1) 40 CFR Part 50 App F Section 1.3.2 2 and 3) Recommendation
Gas Dilution Systems	1/year or after failure of 1 point QC check or performance evaluation	Accuracy \pm 2 %	1,2 and 3) Recommendation based on SO ₂ requirement in 40 CFR Part 50 App A-1 Sec 4.1.2
Detection (FEM/FRMs)			
<i>Noise</i>	NA	<i>0.005 ppm</i>	1) 40 CFR Part 53.23 (b) (definition & procedure) 2) NA 3) 40 CFR Part 53.20 Table B-1
<i>Lower detectable level</i>	1/year	<i>0.01 ppm</i>	1) 40 CFR Part 53.23 (c) (definition & procedure) 2) Recommendation 3) 40 CFR Part 53.20 Table B-1
SYSTEMATIC CRITERIA- NO₂			
<i>Sampler/Monitor</i>	NA	<i>Meets requirements listed in FRM/FEM designation</i>	1) 40 CFR Part 58 App C Section 2.1 2) NA 3) 40 CFR Part 53 & FRM/FEM method list
<i>Standard Reporting Units</i>	<i>All data</i>	<i>ppb (final units in AQS)</i>	1,2 and 3) 40 CFR Part 50 App S Sec 2 (c)
<i>Rounding convention for data reported to AQS</i>	<i>All data</i>	<i>1 place after decimal with digits to right truncated</i>	1, 2 and 3) 40 CFR Part 50 App S Sec 4.2 (a)
<i>Completeness</i>	<i>Annual Standard</i>	\geq 75% hours in year	1) 40 CFR Part 50 App S sec 3.1(b) 2) 40 CFR Part 50 App S sec 3.1(a) 3) 40 CFR Part 50 App S sec 3.1(b)
	<i>1-hour standard</i>	1) 3 consecutive calendar years of complete data 2) 4 quarters complete in each year 3) \geq 75% sampling days in quarter 4) \geq 75% of hours in a day	1) 40 CFR Part 50 App S sec 3.2(b) 2) 40 CFR Part 50 App S sec 3.2(a) 3) 40 CFR Part 50 App S sec 3.2(b) More details in 40 CFR Part 50 App S
<i>Sample Residence Time Verification</i>	1/year	< 20 seconds	1) 40 CFR Part 58 App E, section 9 (c) 2) Recommendation 3) 40 CFR Part 58 App E, section 9 (c)
<i>Sample Probe, Inlet, Sampling train</i>	<i>All sites</i>	<i>Borosilicate glass (e.g., Pyrex®) or Teflon®</i>	1, 2 and 3) 40 CFR Part 58 App E sec 9 (a) FEP and PFA have been accepted as equivalent material to Teflon. Replacement or cleaning is suggested as 1/year and more frequent if pollutant

1) Requirement (NO ₂)	2) Frequency	3) Acceptance Criteria	Information /Action
			load or contamination dictate
Siting	1/year	<i>Meets siting criteria or waiver documented</i>	1) 40 CFR Part 58 App E, sections 2-6 2) Recommendation 3) 40 CFR Part 58 App E, sections 2-6
<i>Precision (using 1-point QC checks)</i>	<i>Calculated annually and as appropriate for design value estimates</i>	90% CL CV ≤ 15%	1) 40 CFR Part 58 App A sec 2.3.1.5 & 3.2.1 2) 40 CFR Part 58 App A sec 4 (b) 3) 40 CFR Part 58 App A sec 4.1.2
<i>Bias (using 1-point QC checks)</i>	<i>Calculated annually and as appropriate for design value estimates</i>	95% CL ≤ ± 15%	1) 40 CFR Part 58 App A sec 2.3.1.5 & 3.2.1 2) 40 CFR Part 58 App A sec 4 (b) 3) 40 CFR Part 58 App A sec 4.1.3
<i>Annual PE Primary QA Organization (PQAO) Evaluation</i>	1/year	<i>95% of audit percent differences fall within the one point QC check 95% probability intervals at PQAO level of aggregation</i>	1) 40 CFR Part 58 App A Section 3.2.2 2) Recommendation 3) 40 CFR Part 58 App A sec 4.1.4 & 4.1.5

NOTE: Polk County Air Quality's SOP may specify more stringent requirements for non-critical criteria or more frequent QA procedures than required by the EPA. The operational and systematic criteria listed in the MQO Table are the Federal minimum criteria only.

APPENDIX A – Forms and Field Sheets

Form 1.....Gas Comparison Spreadsheet
Form 2.....Calibration Field Sheet
Form 3.....Calibration Linear Regression
Form 4.....Calibration Converter Efficiency
Form 5.....Audit Field Sheet
Form 6.....Audit Converter Efficiency
Form 7.....Verification Field Sheet
Form 8.....Verification Converter Efficiency
Form 9.....Corrective Action Form

Form 1: Gas Comparison Spreadsheet

Date	02/23/16
Time	10:00AM
Site	Carpenter
Pollutant	NOX
Operator	JM

Gases

Standard Cylinder Conc.	10.71	PPM
Serial Number	CC12458	
Expiration Date	03/11/16	
Candidate Cylinder Conc.	10.17	PPM
Serial Number	ND45562	
Expiration Date	12/28/18	

Instruments

Gas Calibrator	Thermo 146i	1126649800
Last Verification	2/17/2016	
Monitor	Thermo TL-42i	1117248572
Last Calibrated	4/21/2015	

EXTERNAL ZERO TEST

	<i>AIR</i>	<i>GAS</i>	<i>Known Value</i>	<i>Instrument Response</i>
Verification	Zero Air Actual MFC	Gas Pollutant Actual MFC	NO	NO
	SCCM	SCCM	PPB	PPB
Standard Zero	7995	0.000	0.0	0.0
Candidate Zero	7995	0.000	0.0	0.0

VERIFICATION

	<i>AIR</i>	<i>GAS</i>	<i>Known Value</i>	<i>Instrument Response</i>	<i>% Difference</i>	<i>Pass</i>
Verification	Zero Air Actual MFC	Gas Pollutant Actual MFC	NO	NO	NO	NO
	SCCM	SCCM	PPB	PPB	PPB	PPB
Standard Precision 20	12959	24.24	20.0	19.5		
Candidate Precision 20	12959	25.56	20.0	19.0	2.6	TRUE

Standard Span 225	4019	86.34	225.0	219.8		
Candidate Span 225	4013	90.95	225.0	214.0	2.7	TRUE

Average Percent Diffenece	Is Average Percent Diffence ±4% ?
2.7	TRUE

Is Candidate Gas OK ?
TRUE

Form 2: Calibration Field Sheet

NO-NO₂-NO_x Calibration Sheet

Routine Site Information

Date/Time: _____
 Operator: _____

Analyzer Serial #: _____
 Date of last Calibration: _____
 Calibrator Serial #: _____
 Date of last Calibration: _____
 Date of last Verification: _____
 Date of last Audit: _____

Prec cylinder #: _____
 Cylinder Expiration Date: _____
 Cylinder > 200 psig (Y or N)
 Cylinder Concentration: _____
 Cylinder NO Concentration: _____
 Cylinder NOX Concentration: _____
 Impurity in Tank: _____
 Zero Air Generator Serial #: _____
 Date of last annual maintenance: _____

Station Observations Made (Y or N)
 Changed Filter? (Y or N)
 Sample Line Checked? (Y or N)

NO Reading (ppm): _____
 NO₂ Reading (ppm): _____
 NO_x Reading (ppm): _____

Date Last Calibration: _____
 Ambient: _____

Site: _____

Make adjustments per service manual

Diagnostic Check		
ALARMS DETECTED:		
Alarm	Value	Corrective Action
Internal Temp (°C) (15 - 45°C)		Consult Manual
Chamber Temp (°C) (48 - 52°C)		Check Reaction Chamber
Cooler Temp (°C) (-25 to -1°C)		Check PMT Cooler
Conv. Temp (°C) (300 - 350°C)		Check NO ₂ to NO Converter
Pressure (200 -450 mm Hg)		Replace Reaction Chamber
Sample Flow (0.75 - 2.0 LPM)		Replace Pump
Ozonator Flow (> 0.050 LPM)		Replace Pump
Leak Check (Flow = 0; Pressure < 250 mmHg)		Locate Leak Replace solenoid valves Replace Capillaries

Note: Refer to Section 2.8 of Polk County Air Quality's Standard Operating Procedures NO-NO₂-NO_x for exact calibration procedures.

Gas A Dilution – NO/NO_x

Zero Air Check

Zero Actual: _____scm

Gas Actual: _____scm

Point 1 NO/NO_x (200 ppb)

Calibrator Actual: _____

Zero Actual: _____scm

Gas Actual: _____scm

Point 2 NO/NO_x (160 ppb)

Calibrator Actual: _____

Zero Actual: _____scm

Gas Actual: _____scm

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC Reading				ESC Reading				ESC Reading			
% diff.				% diff.				% diff.			

Point 3 NO/NO_x (120 ppb)

Calibrator Actual: _____

Zero Actual: _____scm

Gas Actual: _____scm

Point 4 NO/NO_x (80 ppb)

Calibrator Actual: _____

Zero Actual: _____scm

Gas Actual: _____scm

Point 5 NO/NO_x (40 ppb)

Calibrator Actual: _____

Zero Actual: _____scm

Gas Actual: _____scm

	NO	NO ₂	NO _x		NO	NO ₂	NO _x	9)	NO	NO ₂	NO _x
ESC Reading				ESC Reading				ESC Reading			
% diff.				% diff.				% diff.			

GPT – NO₂

Zero Air Check

GPT : _____ 0%

Zero Air Flow: _____scms

Gas Air Flow: _____scms

Point 1 (~200 ppb)

GPT %: _____

Point 2 (~160 ppb)

GPT %: _____

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC Reading				ESC Reading				ESC Reading			
[NO ₂] ACT				[NO ₂] ACT				[NO ₂] ACT			
% diff.				% diff.				% diff.			

Point 3 (~120 ppb)

GPT % : _____

Point 4 (~80 ppb)

GPT %: _____

Point 5 (~40 ppb)

GPT %: _____

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC Reading				ESC Reading				ESC Reading			
[NO ₂] ACT				[NO ₂] ACT				[NO ₂] ACT			
% diff.				% diff.				% diff.			

Coefficients:

	NO BKG (< 15 ppb)	NO _x BKG (< 15 ppb)	PREREACTOR (< 15 PPB)	NO COEF (0.900-1.100)	NO ₂ COEF (0.960-1.100)	NO _x COEF (0.900-1.100)	PMT	COUNTS
Before								
After								

Form 3 – Calibration Linear Regression

NO/NO_y-NO/NO_x 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 acceptable		Pass/Fail
							squares line	distance	
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

NO_x Cal

Known Conc.	Measured Conc.	rpd	slope	intercept	rsq	forecast	distance to least acceptable		Pass/Fail
							squares line	distance	
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 acceptable		Pass/Fail
							squares line	distance	
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 4 – Calibration Converter Efficiency

NO₂ Calibration Spreadsheet

Station:	Carpenter	Date:	12/10/2013	Operator:	JVB
Analyzer:	1117248572	Calibrator:	1126649800	Cylinder:	CC12458
Cylinder Conc.	10.71	PPM			
Impurity	0.00	PPM			

Yellow highlight signifies values entered by user.

Green highlight signifies NO₂ Actual for precision data

NO, NO_x CALIBRATION

Calibration	AIR		GAS		Known Value		Instrument Response		
	Zero Air MFC		Pollutant MFC		NO	NO _x	NO	NO _x	NO ₂
	SCCM		SCCM		PPB	PPB	PPB	PPB	PPB
Zero	7999		0.00		0.0	0.0	0.0	0.0	0.0
Point 5 (40 PPB)	8431		31.76		40.2	40.2	40.1	40.4	0.2
Point 4 (80 PPB)	8403		63.48		80.3	80.3	80.7	81.1	0.2
Point 3 (120 PPB)	5631		63.84		120.1	120.1	118.0	118.7	0.4
Point 2 (160 PPB)	4537		68.70		159.8	159.8	159.6	160.2	0.3
Point 1 (200 PPB)	4511		85.91		200.2	200.2	200.4	201.1	0.6
Ozone Lamp 0%	4470		85.95						

NO₂ CALIBRATION (GPT)

Calibration points NO ₂	Instrument Response			Corrected Instrument Response and Calculated Concentrations		
	NO	NO _x	NO ₂	[NO]ORIG	[NO]REM	[NO ₂]ACT
	PPB	PPB	PPB	PPB	PPB	PPB
Ozone Lamp 0%	228.7	228.5	-0.2	229.1	229.1	0.0
Point 5 (40 PPB)	181.0	228.1	47	229.1	181.4	47.7
Point 4 (80 PPB)	145.4	228.5	83.1	229.1	145.7	83.4
Point 3 (120 PPB)	106.0	229.1	123.2	229.1	106.3	122.8
Point 2 (160 PPB)	66.5	229.0	162.6	229.1	66.7	162.4
Point 1 (200 PPB)	30.8	229.8	199.3	229.1	31.0	198.1

	SLOPE	INT.
NO	0.9990	-0.1768
NO _x	1.0024	-0.0631

NO₂ CONVERTER EFFICIENCY TEST

Pollutant Level	[NO ₂]Actual	[NO _x]ORIG	[NO _x]REM	[NO ₂]CONV	Converter Efficiency	Is Converter Efficiency 1 ±0.04 ?
	PPB	PPB	PPB	PPB		
Point 5 (40 PPB)	47.7	228.0	227.6	47.3	99.2%	TRUE
Point 4 (80 PPB)	83.4	228.0	228.0	83.4	100.0%	TRUE
Point 3 (120 PPB)	122.8	228.0	228.6	123.4	100.5%	TRUE
Point 2 (160 PPB)	162.4	228.0	228.5	162.9	100.3%	TRUE
Point 1 (200 PPB)	198.1	228.0	229.3	199.4	100.7%	TRUE

Converter Efficiency (from slope)	Is Converter Efficiency 1 ±0.04 ?
101.0%	TRUE

Form 5 – Audit Field Sheet
NO-NO₂-NO_x Audit Sheet

Site: _____

Routine Site Information

Date/Time: _____
 Quarter/Year: _____
 Operator/Auditor: _____
 Analyzer Serial #: _____
 Date of last calibration: _____
 Audit Calibrator Serial #: _____
 Date of last calibration: _____
 Cylinder Gas Serial #: _____
 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: _____
 Tank Expiration: _____
 Station Observations Made? (Y or N) _____
 Moisture Trap Checked? (Y or N) _____
 Sample Line Checked? (Y or N) _____

Record NO BKG _____
 Record NO_x BKG: _____
 Record NO Coef: _____
 Record NO₂ Coef: _____
 Record NO_x Coef: _____
 Reading NO: _____
 Reading NO₂: _____
 Reading NO_x: _____
 Date Last _____
 Zero/Prec/SpanCheck _____ Days _____
 Ambient: _____

Make adjustments per service manual

Diagnostic Check		
ALARMS DETECTED:		
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

Zero Air Check

Calibrator Actual: _____
 Zero Flow: _____ sccms
 Gas Flow: _____ sccms

Pt. 4 (3-5 ppb)

Calibrator Actual: _____
 Zero Flow: _____ sccms
 Gas Flow: _____ sccm

Pt. 3 (6-19 ppb)

Calibrator Actual: _____
 Zero Flow: _____ sccms
 Gas Flow: _____ sccm

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC Reading				ESC Reading				ESC Reading			
% diff				% diff				% diff			

If there is no reason to believe the results are incorrect or not representative of the analyzers performance, the cause for the audit failure must be investigated and corrected. An investigation initiated by audit results that are outside the above limits may require the invalidation of data.

Pt. 2 (20-100 ppb)

Calibrator Actual: _____
 Zero Flow: _____ sccm
 Gas Flow: _____ sccm

Pt. 1 (110-250 ppb)

Calibrator Actual: _____
 Zero Flow: _____ sccm
 Gas Flow: _____ sccm

	NO	NO ₂	NO _x		NO	NO ₂	NO _x	
ESC Reading				ESC Reading				ESC Reading
% diff				% diff				% diff

$$\frac{ESC(Y) - Calibrator(X)}{Calibrator(X)} \times 100 = \% \text{ Difference}$$

Gas A Dilution – NO/NO_x

Zero Air Check

GPT %: _____
 Zero Air Flow: _____scms
 Gas Air Flow: _____scms

Pt. 4 (3-5 ppb)

GPT %: _____

Pt. 3 (6-19 ppb)

GPT %: _____

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC Reading				ESC Reading				ESC Reading			
NO ₂ Actual				NO ₂ Actual				NO ₂ Actual			
% diff				% diff				% diff			

Zero Air Check

GPT %: _____
 Zero Air Flow: _____scms
 Gas Air Flow: _____scms

Pt. 2 (20-100 ppb)

GPT %: _____

Pt. 1 (110-250 ppb)

GPT %: _____

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC Reading				ESC Reading				ESC Reading			
NO ₂ Actual				NO ₂ Actual				NO ₂ Actual			
% diff				% diff				% diff			

If there is no reason to believe the results are incorrect or not representative of the analyzers performance, the cause for the audit failure must be investigated and corrected. An investigation initiated by audit results that are outside the above limits may require the invalidation of data.

$$NO_{2(Actual)} = (NO_{ORIG} - NO_{REM}) + NO_{2(imp)}$$

Where:

NO_{2Actual} = Actual NO₂ concentration

NO_{2(imp)} = Calculated NO impurities

NO_{ORIG} = Original NO data logger reading, when NO₂ is set to zero

NO_{REM} = Remaining NO data logger reading, after addition of O₃

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

Where:

F_{NO} = NO Flow

F_O = Ozone Flow

F_D = Dilution Flow

Form 6 – Audit Converter Efficiency

NO₂ Audit Spreadsheet

Station:	Carpenter	Date:	12/10/2015	Operator:	JVB
Analyzer:	1117248572	Calibrator:	1126649799	Cylinder:	CC28174
Cylinder Conc.	11.19	PPM			
Impurity	0.00	PPM			

Yellow highlight signifies values entered by user.
 Green highlight signifies NO₂ Actual for precision data

NO, NO_x AUDIT

Audit	AIR		GAS		Known Value		Instrument Response		
	Zero Air MFC	Pollutant MFC	NO	NO _x	NO	NO _x	NO	NO _x	NO ₂
	SCCM	SCCM	PPB	PPB	PPB	PPB	PPB	PPB	PPB
Zero	7987	0.00	0.0	0.0	0.0	0.0	-0.4	-0.4	
Point 4 (8-19 PPB)	15960	21.46	15.0	15.0	14.2	14.1	-0.1		
Point 3 (20-49 PPB)	17930	56.17	34.9	34.9	33.6	33.5	-0.2		
Point 2 (50-100 PPB)	9929	62.47	70.0	70.0	68.0	68.2	0.1		
Point 1 (110-300 PPB)	5622	73.87	145.1	145.1	142.3	143.3	0.7		
Ozone Lamp 0%-Low	9929	62.47							
Ozone Lamp 0%-High	4519	82.24							

NO2 AUDIT (GPT)-LOW RANGE

Audit Points NO2	Instrument Response			Corrected Instrument Responses and Calculated Concentrations		
	NO	NOx	NO2	[NO]ORIG	[NO]REM	[NO2]ACT
	PPB	PPB	PPB	PPB	PPB	PPB
Ozone Lamp 0%	66.7	66.3	-0.5	68.4	68.4	0.0
Point 4 (3-5 PPB)	62.8	66.9	4	68.4	64.4	4.0
Point 3 (6-19 PPB)	50.0	67.0	16.8	68.4	51.4	17.0

NO2 AUDIT (GPT)-HIGH RANGE

Audit Points NO2	Instrument Response			Corrected Instrument Responses and Calculated Concentrations		
	NO	NOx	NO2	[NO]ORIG	[NO]REM	[NO2]ACT
	PPB	PPB	PPB	PPB	PPB	PPB
Ozone Lamp 0%	199.8	197.9	-2	203.9	203.9	0.0
Point 2 (50-100 PPB)	124.0	198.8	74.5	203.9	126.7	77.2
Point 1 (110-300 PPB)	54.7	199.3	144.5	203.9	56.1	147.7

LOW GAS

	SLOPE	INT.
NO	0.9822	-0.4481
NOx	0.9914	-0.8137

NO2 CONVERTER EFFICIENCY TEST

Pollutant Level	[NO ₂]Actual PPB	[NO _x]ORIG PPB	[NO _x]REM PPB	[NO ₂]CONV PPB	Converter Efficiency	Is Converter Efficiency 1 ±0.04 ?
Point 4 (3-5 PPB)	4.0	67.7	68.3	4.6	115.2%	FALSE
Point 3 (6-19 PPB)	17.0	67.7	68.4	17.7	104.2%	TRUE
Point 2 (50-100 PPB)	77.2	200.4	201.4	78.1	101.2%	TRUE
Point 1 (110-300 PPB)	147.7	200.4	201.9	149.1	101.0%	TRUE

Converter Efficiency (from slope)	Is Converter Efficiency 1 ±0.04 ?
100.5%	TRUE

Form 7 – Verification Field Sheet

NO-NO₂-NO_x Verification Sheet

Site: _____

Routine Site Information

Date/Time: _____
 Operator: _____

Analyzer Serial #: _____
 Date of last Calibration: _____
 Calibrator Serial #: _____
 Date of last Calibration: _____
 Date of last Verification: _____
 Date of last Audit: _____

Prec cylinder #: _____
 Cylinder Expiration Date: _____
 Cylinder > 200 psig (Y or N) _____
 Cylinder Concentration: _____
 Cylinder NO Concentration: _____
 Cylinder NO_x Concentration: _____
 Impurity in Tank: _____
 Zero Air Generator Serial #: _____
 Date of last annual maintenance: _____

Station Observations Made (Y or N) _____
 Changed Filter? (Y or N) _____
 Sample Line Checked? (Y or N) _____

Record NO BKG _____
 Record NO_x BKG: _____
 Record PreReactor BKG: _____
 Record NO Coef: _____
 Record NO₂ Coef: _____
 Record NO_x Coef: _____
 Reading NO: _____
 Reading NO₂: _____
 Reading NO_x: _____

Date Last _____
 Zero/Prec/SpanCheck _____ Days _____
 Ambient: _____

Diagnostic Check		
ALARMS DETECTED:		
Alarm	Value	Corrective Action
Internal Temp (°C) (15 - 45°C)		Consult Manual
Chamber Temp (°C) (48 - 52°C)		Check Reaction Chamber
Cooler Temp (°C) (-25 to -1°C)		Check PMT Cooler
Conv. Temp (°C) (300 - 350°C)		Check NO ₂ to NO Converter
Pressure (200 - 450 mm Hg)		Replace Reaction Chamber
Sample Flow (0.75 - 2.0 LPM)		Replace Pump
Ozonator Flow (> 0.050 LPM)		Replace Pump

Make adjustments per service manual

Gas A Dilution – NO/NO_x

Zero Air Check

Zero Air Flow: _____ sccms
 Gas Air Flow: _____ sccms

Precision Check (~20 ppb)

Calibrator Actual: _____
 Zero Air Flow: _____ sccm
 Gas Air Flow: _____ sccm

Span Check (~200 ppb)

Calibrator Actual: _____
 Zero Air Flow: _____ sccm
 Gas Air Flow: _____ sccm

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC reading				ESC reading				ESC reading			
Drift				% diff. Drift				% diff. Drift			

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 $\pm 15\%$, or if the zero drift is ≥ 3.0 ppb, or if the span drift is $\pm 10\%$. See Section 2.8 for calibration procedures.

Zero Drift = Current Data Logger Reading – Calibrator Reading

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

GPT – NO₂

Zero Air “Orig” Check

GPT % : ___0%___

Zero Air Flow: _____sccms

Gas Air Flow: _____sccms

Precision Check (~20 ppb)

GPT %: _____

Span Check (~200 ppb)

GPT %: _____

	NO	NO ₂	NO _x		NO	NO ₂	NO _x		NO	NO ₂	NO _x
ESC reading				ESC reading				ESC reading			
Actual NO ₂ :				Actual NO ₂ :				Actual NO ₂ :			
Drift				% diff.				% diff. Drift			

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 ≥3.0 ppb, or if the span drift is ± 10%. See Section 2.8 for calibration procedures.

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

Where:

NO_{2Actual} = Actual NO₂ 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 O₃

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

Where:

F_{NO} = NO Flow

F_O = Ozone Flow

F_D = Dilution Flow

Form 8 – Verification Converter Efficiency

NO₂ Verification Spreadsheet

Station:	Carpenter	Date:	12/27/2013	Operator:	JVB
Analyzer:	1117248572	Calibrator:	1126649800	Cylinder:	CC12458
Cylinder Conc.	10.71	PPM			
Impurity	0.00	PPM			

Yellow highlight signifies values entered by user.
 Green highlight signifies NO₂ Actual for precision data

NO, NO_x VERIFICATION

Verification	AIR	GAS	Known Value		Instrument Response		
	Zero Air MFC	Pollutant MFC	NO	NO _x	NO	NO _x	NO ₂
	SCCM	SCCM	PPB	PPB	PPB	PPB	PPB
Zero	8005	0.00	0.0	0.0	-0.1	0.3	0.4
Precision	10499	19.56	19.9	19.9	19.5	20.2	0.6
Span (90% URL)	4511	85.91	200.2	200.2	195.4	197.6	2.0
Ozone Lamp 0%	3861	84.11					

NO₂ VERIFICATION (GPT)

Verification points NO ₂	Instrument Response			Corrected Instrument Responses and Calculated Concentrations		
	NO	NO _x	NO ₂	[NO]ORIG	[NO]REM	[NO ₂]ACT
	PPB	PPB	PPB	PPB	PPB	PPB
Ozone Lamp 0%	225.8	226.0	0.3	231.3	231.3	0.0
Precision	202.0	226.1	24.1	231.3	206.9	24.4
Span	37.1	227.0	190.1	231.3	38.0	193.3

	SLOPE	INT.
NO	0.9763	-0.0074
NO _x	0.9851	0.4326

NO₂ CONVERTER EFFICIENCY TEST

Pollutant Level	[NO ₂]Actual	[NO _x]ORIG	[NO _x]REM	[NO ₂]CONV	Converter Efficiency	Is Converter Efficiency 1 ±0.04 ?
	PPB	PPB	PPB	PPB		
Precision	24.4	229.0	229.1	24.5	100.4%	TRUE
Span	193.3	229.0	230.0	194.3	100.5%	TRUE

Converter Efficiency (from slope)	Is Converter Efficiency 1 ±0.04 ?
100.5%	TRUE

NOTE: All formulas for calculating converter efficiency come from: EPA Quality Assurance Guidance Document 2.3, Reference Method for the Determination of Nitrogen Dioxide in the Atmosphere (Chemiluminescence), February, 2002, Draft.

Form 9 – 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: _____