# Standard Operation Procedures for Near-Road NO<sub>2</sub>

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 TF	RACE LEVEL NO-NO <sub>2</sub> -NO <sub>X</sub> MONITORING
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# 3.0 STANDARD OPERATING PROCEDURE FOR NEAR-ROAD NO<sub>2</sub>

# 3.1 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 near-road NO<sub>2</sub> monitoring 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.

# 3.2 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 for near-road microscale NO<sub>2</sub> monitoring. 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 42*i* analyzers operated by Polk County Air Quality personnel.

# 3.3 References

- 3.3.1 Thermo Scientific, Model 42*i* Trace Level, Chemiluminescence NO-NO<sub>2</sub>-NO<sub>x</sub> Analyzer, Instruction Manual. Part Number 102855-00, December 20, 2007.
- 3.3.2 Thermo Scientific, Model 146i, Dynamic Gas Calibrator, Instruction Manual. Part Number 102482-00, January 30, 2008.
- 3.3.3 EPA Quality Assurance Guidance Document 2.3, Reference Method for the Determination of Nitrogen Dioxide in the Atmosphere (Chemiluminescence), February, 2002, Draft.
- 3.3.4 EPA-600/4-75-003 December 1975, Technical Assistance Document for the Chemiluminescence Measurement of Nitrogen Dioxide.
- 3.3.5 EPA-600/R-12/531 May, 2012, EPA Traceability Protocol for Assay and Certification of Gaseous Calibration Standards.
- 3.3.6 EPA-454/B-13-003, May 2013. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II. Ambient Air Quality Monitoring Program.
- 3.3.7 EPA-454/B-13-003, July 2014. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II. Ambient Air Quality Monitoring Program, Appendix D
- 3.3.8 40 Code of Federal Regulations (CFR) Part 50, Appendix F, Measurement Principle and Calibration Procedure for the Measurement of  $NO_X$  in the Atmosphere.
- 3.3.9 40 Code of Federal Regulations (CFR) Part 58, Appendix A, Quality Assurance Requirements for State and Local Air Monitoring Stations (Slams).
- 3.3.10 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 Assessement Monitoring Stations (PAMS).
- 3.3.11 40 Code of Federal Regulations (CFR) Part 58, Appendix E, Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring.
- 3.3.12 EPA-454/B-12-002, June 2012, Near-Road NO<sub>2</sub> Monitoring Technical Assistance Document. By: Nealson Watkins and Dr. Richard Baldauf.

# 3.4 Introduction

In February of 2010, the EPA revised the minimum monitoring requirements for the nitrogen dioxide (NO<sub>2</sub>) monitoring network in support of a new 1-hour NO<sub>2</sub> national Ambient Air Quality Standards (NAAQS) (75 FR 6474, Feb. 9, 2010). In support of that standard, state and local agencies with a CBSA greater than 500,000 were required to install a near-road NO<sub>2</sub> monitoring station where peak hourly NO<sub>2</sub> concentrations are most likely to occur. The U.S. OMB's official name for the Des Moines area CBSA is the Des Moines-West Des Moines MSA. The counties that make up this MSA are Polk, Dallas, Warren, Madison, and Guthrie. According to the 2010 census data from the U.S Census Bureau, the population of the Des Moines-West Des Moines MSA is 569,633.

Measurements of nitrogen dioxide (NO<sub>2</sub>) in ambient air are based on the principle that nitric oxide (NO) and ozone (O<sub>3</sub>) react to produce a characteristic luminescence with an intensity linearly proportional to the NO concentration. Infrared light emission results when electronically excited NO<sub>2</sub> molecules decay to lower energy states:

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

Nitrogen dioxide (NO<sub>2</sub>) must first be transformed into NO before it can be measured using the chemiluminescent reaction. NO<sub>2</sub> is converted to NO by a molybdenum NO<sub>2</sub> to NO converter heated to 325°C.

Ambient air is drawn into the  $NO-NO_2-NO_x$  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. The sample then flows through the converter output valve and a flow sensor to the prereactor solenoid valve.

The prereactor solenoid valve directs the sample either to the reaction chamber, where it mixes with ozone to give an NO reading, or to the prereactor where it reacts with ozone prior to the reaction chamber giving the dynamic zero reading for the analyzer. 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-O<sub>3</sub> 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  $\leq 2$  minutes and the dynamic parameter is  $\geq 2.75$  ppm-min, in accordance with formulas given to the analyzer operator's manual. Section 8-4, Thermo Scientific 146*i* Instruction Manual.

# 3.5 Health and Safety Warning/Precautions

Only properly trained personnel should perform TL-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 TL-42*i*. Gas cylinders can sometimes contain pressures as high as 2000 pounds per square inch (psi). Handling of cylinders must be

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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.

# 3.6 Sampler Setup

# 3.6.1 Site Selection

The purpose of a near-road NO<sub>2</sub> monitoring station is to characterize the maximum expected hourly NO<sub>2</sub> concentration due to mobile source emissions on major roadways. Therefore, the most important spatial scale for near-road NO<sub>2</sub> monitoring station is microscale. Microscale represents areas in close proximity to major roadways or point and area sources, and extending up to approximately 100 meters. Near-road NO<sub>2</sub> monitoring stations are required to be within 50 meters of target road segments

For more detailed information concerning site selection for near-road NO<sub>2</sub> 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."

# **3.6.1.1** The Monitoring Station

The structure housing the near-road NO<sub>2</sub> monitoring equipment is 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<sub>2</sub>-NO<sub>x</sub> 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.

# 3.6.1.2 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 2-7 meters above ground level.
- The monitor probe shall be as near as practicable to the outside nearest edge of the traffic lanes of the target road segment; but shall not be located at a distance greater than 50 meters in the horizontal, from the outside nearest edge of the traffic lanes of the target road segment.
- The monitor probe shall have an unobstructed air flow, where no obstacles exist at or above the height of the monitor probe, between the monitor probe and the outside nearest edge of the traffic lanes of the target road segment.
- Trees can provide surfaces for NO<sub>2</sub> 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.
- No trees or shrubs should be located between the probe and the roadway under investigation.

For more detailed information concerning site selection for near-road NO<sub>2</sub> 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.

# 3.6.2 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 it is discovered 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.

# 3.6.3 Equipment Installation

Polk County Air Quality Division will use the Thermo Scientific Model 42*i* Trace Level (TL-42*i*) Chemiluminescence NO-NO<sub>2</sub>-NO<sub>x</sub> Analyzer displayed in Figure 3-1 for sampling nitrogen dioxide. Installation of the TL-42*i* 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 3-1: Thermo Scientific TL-42*i* NO-NO<sub>2</sub>-NO<sub>x</sub> Analyzer



The Thermo TL-42*i* 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  $NO-NO_2-NO_X$  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, installation of a Teflon filter between the ambient sample line and the sample port of the analyzer must be done prior to 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.

# 3.6.4 Programming the TL-42*i*

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<sub>2</sub>-NO<sub>X</sub> 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  $\uparrow$  or  $\downarrow$  buttons to move the cursor to each submenu.

# 3.6.4.1 Range and Units

The Range menu defines the concentration range of the analog outputs. Polk County Air Quality will use the "Single Range Mode" option with the selected range of 0 - 500 ppb for the analyzer.

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To set the range for the instrument, press the **MENU** button to access the Main Menu. Press the  $\downarrow$  button until the cursor is on "NO Range." Press **ENTER** to display the NO Range Menu, and select **RANGE**. Use the  $\uparrow$  or  $\downarrow$  buttons to scroll through the preset ranges. Select "500" and press **ENTER**. Press **MENU** to return to the Range Menu. Repeat procedures to set NO<sub>2</sub> Range and NO<sub>X</sub> Range.

The Gas Units Screen defines how the NO-NO<sub>2</sub>-NO<sub>X</sub> concentration reading is expressed. From the Range Menu, select **GAS UNITS** to display the Gas Units screen. Use the  $\downarrow$  button to select "PPB" and press **ENTER**. Press **MENU** twice to return to the Run Screen.

# 3.6.4.2 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. Polk County Air Quality will use the setting of 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  $\downarrow$  button to scroll to **AVERAGING TIME** and press **ENTER**. From the Averaging Time Screen, use the  $\uparrow$  **or**  $\downarrow$  buttons to scroll through the preset ranges. Press **ENTER** to select the desired averaging time. Press **RUN** to return to the Run Screen.

# **3.6.4.3** 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 TL-42*i* with the temperature compensation set to ON.

From the Main Menu, use the \$\psi\$ button to scroll to **INSTRUMENT CONTROLS** and press **ENTER**. From the Instrument Controls Screen, use the \$\psi\$ 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.

# 3.6.4.4 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 300 mmHg. **Polk County Air Quality will run the TL-42***i* with the pressure compensation set to ON, so the first line of the display represents the current pressure in the reaction chamber with an acceptable range of 200-450 mmHG. The displayed pressure is measured in pre-reactor mode.

From the Main Menu, use the \$\psi\$ button to scroll to INSTRUMENT CONTROLS and press ENTER. From the Instrument Controls Screen, use the \$\psi\$ 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.

# **3.6.4.5** 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.

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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.

### 3.6.4.6 Clock Set

To set the correct time and date on the instrument, press **MENU** to return to the Main Menu. Use the  $\downarrow$  button to scroll to **INSTRUMENT CONTROLS** and press **ENTER**. Use the  $\downarrow$  buttons to scroll to **DATE/TIME** and press **ENTER**. The date and time should be set to the data logger time. Use the  $\rightarrow$  button to select: year, month, day, hour, minutes, or seconds. Use the  $\uparrow$  or  $\downarrow$  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 1 minute.

# **3.6.4.7 Auto Mode**

The Auto/Manual Mode screen allows selection of the automatic mode (NO/NO<sub>X</sub>), NO Mode (manual NO), or NO<sub>X</sub> mode (manual NO<sub>X</sub>). **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, NO<sub>2</sub>, and NO<sub>X</sub> concentrations are determined.

To set the TL-42i to Auto Mode, press **MENU** to return to the Main Menu. Use the  $\downarrow$  button to scroll to **INSTRUMENT CONTROLS** and press **ENTER**. Use the  $\downarrow$  buttons to scroll to **AUTO/MANUAL MODE** and press **ENTER**. Use the  $\downarrow$  buttons to scroll to **NO/NO**<sub>X</sub> **MODE** and press **ENTER**.

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

# 3.7 Standards

A cylinder containing 20 to 40 ppm NO in N<sub>2</sub> with less than 1.0 ppm NO<sub>2</sub> is used as the concentration standard. The cylinder must be traceable to a National Institute of Standards and Technology (NIST) NO in N<sub>2</sub> Standard Reference Material or NO<sub>2</sub> Standard Reference Material. Procedures for certifying the NO cylinder (working standard) against a NIST traceable NO or NO<sub>2</sub> standard and for determining the amount of NO<sub>2</sub> 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<sub>2</sub> 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. 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. 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 NO<sub>2</sub> 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.

# 3.8 Calibrations

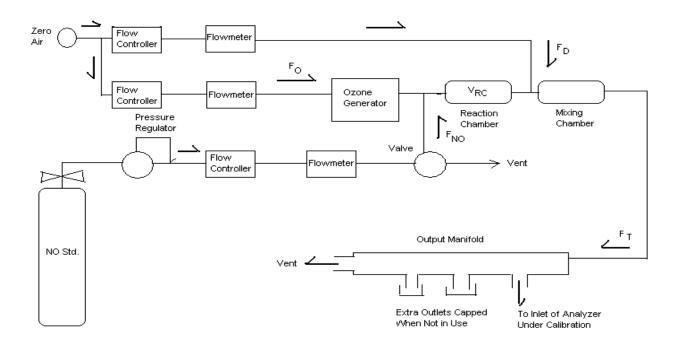
The Thermo Scientific Model TL-42i NO-NO<sub>2</sub>-NO<sub>x</sub> monitor is calibrated whenever the following occurs: installation of a new monitor, instrument repair, when a zero/precision/span check fail to meet acceptance criteria limits, after 6 months has expired since the last calibration, or more frequently at the field operator's discretion.

The Purpose of the calibration is to determine the NO,  $NO_2$  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  $NO_2$ .

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

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Figure 3-2: Gas Phase Titration System



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<sub>2</sub>-NO<sub>x</sub> 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.

# 3.8.1 Calibration Procedures for the TL-42i using the 146i Calibrator

# 3.8.1.1 Pre-Calibration

Change the averaging time to 60 seconds. From the Main Menu, use the  $\downarrow$  button to scroll to **AVERAGING TIME** and press **ENTER**. From the Averaging Time Screen, use the  $\uparrow$  **or**  $\downarrow$  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.

# 3.8.1.2 Calibration of $NO/NO_x$

The GPT requires the use of the  $NO/NO_x$  channel of the analyzer to determine the amount of  $NO_2$  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  $\leftarrow$  and  $\rightarrow$  buttons to select desired **GAS** and press **ENTER**. Use the  $\downarrow$  button to scroll down to **SPAN**. Use the  $\leftarrow$  and  $\rightarrow$  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<sub>2</sub>, and NO<sub>X</sub> 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 PREREACTOR 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<sub>X</sub> BACKGROUND and press ENTER. Press ENTER to set the NO<sub>X</sub> 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 500 ppb (i.e. 400 ppb). Allow the site analyzer to stabilize for a minimum of 15 minutes and record all data logger readings from the NO, NO<sub>2</sub>, and NO<sub>X</sub> 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**. Scroll to **CALIBRATE NO COEFFICIENT** and press **ENTER**. Use the  $\leftarrow$  and  $\rightarrow$  buttons to move the cursor left and right. Use the  $\downarrow$  or  $\uparrow$  buttons to increment and decrement the digit to the expected NO concentration and press **ENTER**.

Repeat to calibrate the NO<sub>X</sub> concentration.

NOTE: The NO,  $NO_X$ , and Prereactor Background coefficients should be less that 15 ppb. The NO and  $NO_X$  span coefficients should always fall between 0.900 and 1.100, and the  $NO_2$  span coefficient should be between 0.960 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 300 ppb, SPAN 3 is set to 200 ppb, SPAN 4 is set to 100 ppb, and SPAN 5 is set to 50 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<sub>2</sub>, and NO<sub>X</sub> channels.

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Repeat for SPAN 2 - 5, respectively. Record all information on the Calibration Field Sheet and Linear Regression Spreedsheet, Forms 2 and 3, Appendix A.

# 3.8.1.3 Calibration of NO<sub>2</sub>

On the main screen of the 146*i* Calibrator, use the  $\downarrow$  button to scroll to the third line. Use the  $\leftarrow$  and  $\rightarrow$  buttons to scroll to **OZON MAN**.

Note: Check and make sure that the second line is set to 450 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<sub>2</sub>, and NO<sub>X</sub> channels. NO<sub>2</sub> should stabilize out at approximately 0 ppb. NO/NO<sub>x</sub> should stabilize out at approximately 450 ppb. Record all information in the site log.

At this point the zero NO<sub>2</sub> 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<sub>2</sub> reading in the range of 400 ppb. Allow the site analyzer to stabilize and record all data logger readings from the NO, NO<sub>2</sub>, and NO<sub>X</sub> 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. 400 ppb). Using the NO calibration relationship determined in Section 3.8.1.2, correct  $NO_{ORIG}$  and  $NO_{REM}$  for slope and intercept prior to calculating  $NO_{2(ACTUAL)}$ .

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

Where:

 $NO_{orig} = Original \ NO \ data \ logger \ reading, \ when \ NO_2 \ is set to zero \ NO_{rem} = Final \ NO \ data \ logger \ reading, \ when \ NO_2 \ is \ approx. 400 \ ppb \ NO_{2(imp)} = Conc. \ of \ NO_2 \ impurity \ in \ standard \ NO \ cylinder$ 

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 NO<sub>2</sub> impurities in the NO concentration standard are determined by:

$$NO_{2(imp)} = (F_{NO} (sccm))(Impurities from cylinder gas (ppb))$$
  
 $F_{NO} + F_{O} + F_{D}$ 

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 146*i* calibrator represents the sum of the dilution air and the flow past the ozone lamp.

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**. Scroll to **CALIBRATE NO<sub>2</sub> COEFFICIENT** and press **ENTER**. Use the  $\leftarrow$  and  $\rightarrow$  buttons to move the cursor left and right. Use the  $\downarrow$  or  $\uparrow$  buttons to increment and decrement the digit to the expected NO<sub>2</sub> concentration. Press **ENTER** to calibrate the analyzer to the desired NO<sub>2</sub> 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 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.

Following the calibration, a check will be done to ensure that the analyzer can pass a precision check with a 4% difference.

Record all information in the site log.

# 3.8.2 Converter Efficiency $(NO_2)$

During a calibration, the converter efficiency will be determined for each  $NO_2$  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 Calibration Converter Efficiency Spreadsheet to determine if the calibration passes, Form 4, Appendix A.

For more detailed information concerning calibration of a Thermo Scientific 42*i* NO-NO<sub>2</sub>-NO<sub>x</sub> 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*-TL, Cheimluminescence NO-NO<sub>2</sub>-NO<sub>x</sub> Analyzer.

# 3.9 Quality Control

# 3.9.1 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 TL-42i NO-NO<sub>2</sub>-NO<sub>x</sub> analyzer.

# 3.9.2 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 will be maintained by Polk County Air Quality and will be archived for future reference.

# 3.9.3 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<sub>x</sub> concentrations generated by the station 146*i* calibrator. The deviation between the "indicated" value of the analyzer and the "actual" value of the calibrator is then determined.

Zero air and  $NO/NO_x$  concentrations of 40-70 ppb (the precision) and 350-450 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  $\geq$  3.0 ppb, or if the span drift is  $\pm$  10%. Acceptance criteria can be found in Table 3-2: 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.

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 3.13.4.

# 3.9.4 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 "call" the monitor site and examine the data recorded at the monitoring station (i.e. NO-NO<sub>2</sub>-NO<sub>x</sub> 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<sub>2</sub> 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.

# 3.10 Equipment, Maintenance and Trouble Isolation

The equipment used to calibrate, audit, perform bi-weekly zero/precision/span checks and monitor near-road  $NO_X$  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.

# **3.10.1 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.

# 3.10.1.1 Analyzer Leak Check

An analyzer leak check should be performed on a yearly basis, or as needed for trouble shooting purposes. To perform an analyzer leak check, 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  $\downarrow$  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 analyzer. Check to see that all fittings are tight, and none of the lines are cracked or broken.

# 3.10.1.2 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 the sample line 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 above the roof, and repeat the check. If a second leak check fails, look for the leak and/or replace sample line if needed.

# 3.10.1.3 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.

# 3.10.1.4 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.

# 3.10.1.5 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.

# 3.10.1.6 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-NO<sub>2</sub>-NO<sub>x</sub> concentrations due to contamination in the line, or every two years.

# **3.10.2** 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-42*i*, Chemiluminescence NO-NO<sub>2</sub>-NO<sub>x</sub> Analyzer Instruction Manual and compile personal notes on troubleshooting as they gain experience with the instrument. The operator is encouraged to contact Thermo Scientific technical support at 1-866-282-0430 when attempting any repairs.

# 3.10.3 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.

# 3.11 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 near-road NO<sub>2</sub> analyzer once per quarter.

# 3.11.1 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)," the near-road NO<sub>2</sub> 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 than the one conducting the routine field measurements and by using audit standards, reference materials, and equipment

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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-42*i* continuous analyzer with known concentrations of NO-NO<sub>2</sub>-NO<sub>X</sub> within the measurement range of the analyzer. The 146*i* Multi-Gas Calibrators can be programmed to generate the desired NO-NO<sub>2</sub>-NO<sub>X</sub> 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, NO<sub>2</sub>, and NO<sub>X</sub> channels. Both the audit calibrator and site analyzer readings should be taken only after a stable response is exhibited by both instruments. The results are recorded on the Quarterly Audit Sheet, Form 5, Appendix A.

The NO<sub>2</sub> Converter Efficiency should also be determined during an audit. Use the Audit 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  $\pm 15\%$  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  $\pm 1.5$  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.

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.

# 3.11.2 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  $NO-NO_2-NO_x$  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<sub>2</sub>-NO<sub>x</sub> and in all other EPA audit programs, which may arise in the future. According to the July 2014 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  $\pm 1.5$  ppb, or  $\pm 15\%$ , whichever is less stringent.

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.

# 3.12 Data Quality Assessment

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

# 3.12.1 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 3.13). Calculations to assess precision are given below and should be used to assess precision on a quarterly basis. 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%; and for bias as an upper 95 percent confidence limit for the absolute bias of 15%.

Precision data 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)".

# 3.12.2 Accuracy

The Polk County Air Quality Program participates in the USEPA performance audit program for NO<sub>2</sub> 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.

# 3.12.3 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.

# 3.13 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 42*i*-TL, Chemiluminescence NO-NO<sub>2</sub>-NO<sub>x</sub> Analyzer, Instruction Manual, and Model 146*i*, Dynamic Gas Calibration System, Instruction Manual.

# 3.13.1 Maintenance Mode for Agilaire Model 8832 Data Loggers

Figure 3-3 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 **CONFIGURE (DATA) CHANNEL** and press **ENTER**. Use the arrow keys to select **PUT CHANNEL IN MAINTENANCE** 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.

# 3.13.2 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.

# 3.13.3 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-42*i* Instruction Manual, Chapter 7-Servicing, and Section 3.10.1 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 the Prereactor, NO and  $NO_x$ . Scroll down and record span coefficients for NO,  $NO_2$ , and  $NO_x$ .

Check the Teflon particulate filter 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 3-1 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	

# 3.13.4 Bi-Weekly Zero, Precision and Span Checks

# **3.13.4.1 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  $\leftarrow$  and  $\rightarrow$  buttons to select desired **GAS** and press **ENTER**. Use the  $\downarrow$  button to scroll down to **SPAN**. Use the  $\leftarrow$  and  $\rightarrow$  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<sub>2</sub>, and NO<sub>x</sub> 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 3.9.3 for acceptable control limits.

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

Where:

 $C_M$  = datalogger concentration, ppb

 $C_{146}$  = Calibrator concentration, ppb

# 3.13.4.2 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 146*i* Calibrator, press the **MENU** button to access the Main Menu Screen. Scroll to **OPERATION** and press **ENTER**. Use the  $\downarrow$  button to scroll down to **SPAN**. Use the  $\leftarrow$  and  $\rightarrow$  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 450 ppb NO/NO<sub>x</sub>). Perform the span check using a cylinder containing 25±2 ppm NO in N<sub>2</sub> with less than 1 ppm NO<sub>2</sub>. 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 TL-42i site analyzer to stabilize for a minimum of 15 minutes and record the resultant data logger readings from the NO, NO<sub>2</sub>, and NO<sub>X</sub> channels on the Verification Field Sheet, Form 7, Appendix A.

Determine the analyzer span drift by comparing the collected span concentration to the actual Calibrator concentration. Refer to Section 3.9.3 for acceptable control limits.

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

Where

 $C_{\scriptscriptstyle M} = {\rm datalogger} \ {\rm concentration}, {\rm ppb}$ 

 $C_{146}$  = Calibrator concentration, ppb

# 3.13.4.3 Precision Check (NO/NO<sub>x</sub>)

At this point the span readings have been recorded, perform the precision level check. Use the  $\leftarrow$  and  $\rightarrow$  buttons to scroll to **SPAN 5** and press **ENTER**. **SPAN 5** has been programmed to produce the precision level approximately 50 ppb NO/NOx.

Allow the TL-42*i* site analyzer to stabilize for a minimum of 15 minutes and record the resultant data logger readings from the NO, NO<sub>2</sub>, and NO<sub>X</sub> channels on the Verification Field Sheet, Form 7, Appendix A.

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

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

Where:

 $C_M$  = datalogger concentration, ppb

 $C_{146}$  = Calibrator concentration, ppb

Acceptance criteria can be found in Table 3-2: 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. If a calibration is necessary, it must be performed after the precision and span checks are finished.

# 3.13.4.4 Zero/Precision/Span Checks (NO<sub>2</sub>)

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  $\downarrow$  button to scroll to the third line. Use the  $\leftarrow$  and  $\rightarrow$  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 450 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 TL-42*i* site analyzer to stabilize for a minimum of 15 minutes and record the resultant data logger readings from the NO, NO<sub>2</sub>, and NO<sub>X</sub> channels on the Zero/Precision/Span Field Sheet. NO<sub>2</sub> should stabilize around 0 ppb. NO/NO<sub>X</sub> channels should stabilize around 450 ppb.

At this point, the zero NO<sub>2</sub> 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<sub>2</sub> reading of approximately 400 ppb. Allow the TL-42*i* site analyzer to stabilize for a minimum of 15 minutes, then record the resultant data logger readings from the NO, NO<sub>2</sub>, and NO<sub>X</sub> channels on the Verification Field Sheet, Form 7, Appendix A.

Use the arrow keys to adjust the ozonator percentage to obtain a  $NO_2$  reading of approximately 50 ppb. Allow the TL-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, Form 7, Appendix A.

Using the NO Verification relationship determined in Section 3.13.4.1-3.13.4.3, correct  $NO_{ORIG}$  and  $NO_{REM}$  for slope and intercept prior to calculating  $NO_{2(ACTUAL)}$ . The  $NO_{2ACTUAL}$  readings are then determined by using the formula:

$$NO_{2Actual} = (NO_{ORIG} - NO_{REM}) + \underbrace{F_{NO} * NO_{2imp}}_{F_{T}}$$

Where:

NO<sub>orig</sub> = Original NO data logger reading

 $NO_{rem} = NO$  data logger reading, after addition of  $O_3$ 

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

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

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

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

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

Where:

NO<sub>2 (Indicated)</sub> = data logger concentration, ppb

Turn off the gas from the cylinder containing 25±2 NO in N<sub>2</sub> with less than 1 ppm NO<sub>2</sub>

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

Refer to Section 3.13.2 to log out from the data logger.

# 3.13.4.5 Converter Efficiency Check

During each bi-weekly verification, the converter efficiency must be determined for the NO<sub>2</sub> 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 Bi-Weekly 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.

# 3.13.5 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  $\ge$ 3.0 ppb, or if the span drift is  $\pm$  10%. See Section 3.8 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 exceed 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 3-2: Measurement Quality Objectives** 

N	iO.	NO	· NO	Valid	ation	Temp	ate
ш				v and	4 11011	I CIIID	

1) Requirement (NO <sub>2</sub> )	2) Frequency	3) Acceptance Criteria	Information /Action
	CRI	TICAL CRITERIA- NO2	
One Point OC Check Single analyzer	1/2 weeks	$\leq \pm 15\%$ (percent difference)	1 and 2) 40 CFR Part 58 Atm 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 $\leq \pm 3.0$ ppb (24 hr) $\leq \pm 5.0$ ppb (>24hr-14 day) Span drift $\leq \pm 10\%$	1 and 2) QA Handbook Volume 2 Section 12.3 3) Recommendation and related to DQO
Converter Efficiency	During multi-point calibrations, span and audit 1/2 weeks	(296%) 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.
	OPERA	ATIONAL 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
Annual Performance Evaluation Single Analyzer	Every site 1/year 25 % of sites quarterly	Percent difference of audit levels $3-10 \le \pm 15\%$ Audit levels $1\&2 \pm 1.5$ ppb difference or $\pm 15\%$	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
Federal Audits (NPAP)	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
Verification/Calibration	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 <sub>2</sub> )	2) Frequency	3) Acceptance Criteria	Information /Action
	daily		
Gaseous Standards	All gas cylinders	NIST Traceable (e.g., EPA Protocol Gas) 50-100 ppm of NO in Nitrogen with < 1 ppm NO <sub>2</sub>	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 EPAAmbient Air Protocol Gas Verification Program 40 CFR Part 58 App A sec 2.6.1
Zero Air/Zero Air Check	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 ± 2 %	1,2 and 3) Recommendation based on SO2 requirement in 40 CFR Part 50 App A-1 Sec 4.1.2
Detection (FEM/FRMs)			
Noise	NA.	0.005 ррт	1) 40 CFR Part 53.23 (b) (definition & procedure) 2) NA 3) 40 CFR Part 53.20 Table B-1
Lower detectable level	1/year	0.01 ppm	1) 40 CFR Part 53.23 (c) (definition & procedure) 2) Recommendation 3) 40 CFR Part 53.20 Table B-1
	SYSTE	EMATIC CRITERIA- NO2	
Sampler/Monitor	NA	Meets requirements listed in FRM/FEM designation	1) 40 CFR Part 58 App C Section 2.1 2) NA 3) 40 CFR Part 53 & FRM/FEM method list
Standard Reporting Units	All data	ppb (final units in AQS)	1,2 and 3) 40 CFR Part 50 App S Sec 2 (c)
Rounding convention for data reported to AQS	All data	1 place after decimal with digits to right truncated	1, 2 and 3) 40 CFR Part 50 App S Sec 4.2 (a)
	Annual Standard	≥ 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)
Completeness	teness 1-hour standard	1) 3consecutive calendars years of complete data 2) 4 quarters complete in each year 3) ≥75% sampling days in quarter 4) ≥ 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
Sample Residence Time Verification	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)
Sample Probe, Inlet, Sampling train	All sites	Borosilicate glass (e.g., Pyrex*) or Teflon*	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 <sub>2</sub> )	2) Frequency	3) Acceptance Criteria	Information /Action
			load or contamination dictate
Siting	1/year	Meets siting criteria or waiver documented	1) 40 CFR Part 58 App E, sections 2-6 2) Recommendation 3) 40 CFR Part 58 App E, sections 2-6
Precision(using 1-point QC checks)	Calculated annually and as appropriate for design value estimates	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
Bias (using 1-point QC checks)	Calculated annually and as appropriate for design value estimates	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
Annual PE Primary QA Organization (PQAO) Evaluation	1/year	95% of audit percent differences fall within the one point QC check 95% probability intervals at PQAO level of aggregation	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
	Audit Field Sheet
Form 6	Audit Converter Efficiency
	Verification Field Sheet
Form 8	Verification Converter Efficiency
	Corrective Action Form

Revision Number: 5

# Form 1 – Gas Comparison Spreadsheet

Date	10/29/14
Time	11:00
Site	Rollins
Pollutant	NOX
Operator	J Bradley

# Gases

	Standard Cylinder Conc.	24.98	PPM
	Serial Number	CB08869	
	Expiration Date	11/27/14	
ı	Canidate Cylinder Conc.	36.10	PPM
ı	Serial Number	CC109875	
	Expiration Date	06/21/17	

# Instruments

Gas Calibrator	Thermo 146i	710920928
Last Verification	6/16/2014	
Monitor	Thermo TL-42i	1125849711
Last Calibrated	6/17/2014	

### EXTERNAL ZERO TEST

EXTERNAL ZERO TEST												
	AIR	GAS	Known Value	Instrument Response								
Verification Zero Air Actual MFC		Gas Pollutant Actual MFC	NO	NO								
	SCCM	SCCM	РРВ	РРВ								
Standard Zero	7998	0.000	0.0	0.0								
Canidate Zero	8003	0.000	0.0	0.1								

# VERIFICATION

	AIR	GAS	Known Value	Instrument Response	% Difference	Pass
Verification	Zero Air Actual MFC	Gas Pollutant Actual MFC	NO	NO	NO	NO2
	SCCM	SCCM	PPB	PPB	PPB	PPB
Standard Precision 50	4418	81.12	50.0	49.7		
Candidate Precision 50	5923	74.78	50.1	48.4	2.7	TRUE

Standard Span 400	4418	81.12	450.4	467.6		
CandidateSpan 400	5923	74.78	450.1	450.8	3.7	TRUE

Avg % Diffenece	Is Avg % Diffence ±4% ?
3.2	TRUE

Is Candidate Gas OK ?	
TRUE	

evised: February 17, 2016 Revision Number: 5

# Form 2 – Calibration Field Sheet

# NO-NO<sub>2</sub>-NO<sub>x</sub> 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):	
NOx Reading (ppm):	
Date Last Calibration:	
Ambient:	

Site:					

	Diagnostic Check ALARMS DETE		
	Alarm	Value	Corrective Action
	Internal Temp (°C) (15-45°C)		Consult Manual
Make adjustments per service manual	Chamber Temp (°C) (48 – 52 °C)		Check Reaction Chamber
	Cooler Temp (°C) (-25 to -1°C)		Check PMT Cooler
stment	Conv. Temp (°C) (300 - 350°C)		Check NO <sub>2</sub> to NO Converter
adju	Pressure (200 - 450 mm Hg)		Replace Reaction Chamber
Make ad	Sample Flow (0.750 – 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 3.9 of Polk County Air Quality's Standard Operating Procedures  $NO-NO_2-NO_x$  for exact calibration procedures.

Revision Number: 5

# Gas A Dilution - NO/NO<sub>x</sub>

 Zero Air Check
 Point 1 NO/NOx (400 ppb)
 Point 2 NO/NOx (300 ppb)

 Calibrator Actual:
 Calibrator Actual:

 Zero Actual:
 sccm
 Zero Actual:
 sccm

 Gas Actual:
 sccm
 Gas Actual:
 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.			

 Point 3 NO/NOx (200 ppb)
 Point 4 NO/NOx (100 ppb)
 Point 5 NO/NOx (50 ppb)

 Calibrator Actual:
 Calibrator Actual:
 Calibrator Actual:

 Zero Actual:
 sccm
 Zero Actual:
 zero Actual:

 Gas Actual:
 sccm
 Gas Actual:
 sccm

	NO	NO <sub>2</sub>	NO <sub>x</sub>		NO	NO <sub>2</sub>	NO <sub>x</sub>		NO	NO <sub>2</sub>	NO <sub>x</sub>
ESC Reading				ESC Reading				ESC Reading			
% diff.				% diff.				% diff.			

# $\underline{GPT - NO_2}$

**Zero Air Check Point 1 (400 ppb) Point 2 (300 ppb) GPT : GPT %: GPT %:** 

0110/0_				OI I	·			GI	1 /0		
	NO	NO <sub>2</sub>	NO <sub>x</sub>		NO	$NO_2$	NO <sub>x</sub>		NO	$NO_2$	$NO_x$
ESC				ESC				ESC			
Reading				Reading				Reading			
[NO <sub>2</sub> ] ACT				[NO <sub>2</sub> ] ACT				[NO <sub>2</sub> ] ACT			
% diff.				% diff.				% diff.			

U	1 1 /0				011/0	·					1 /0			
		NO	$NO_2$	NO <sub>x</sub>		NO	$NO_2$	NO <sub>x</sub>		NO	NO <sub>2</sub>	NO <sub>x</sub>		
	ESC				ESC				ESC					
	Reading				Reading				Reading					
	[NO <sub>2</sub> ] ACT				[NO <sub>2</sub> ] ACT				[NO <sub>2</sub> ] ACT					
	% diff.				% diff.				% diff.					

# **Coefficients:**

	NO BKG (< 15 ppb)	NO <sub>X</sub> BKG (< 15 ppb)	PREREACTOR (< 15 PPB)	NO <sub>2</sub> COEF (0.960-1.100)	 PMT	COUNTS
Before						
After						

Form 3 – Calibration Linear Regression

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

Analyzer: THERMO 42i Serial No.: 1207251984

Site: Carpenter

Full Scale: 500 ppb ppb

1% of Full Scale: 5 Date:

June 21, 2012

Operator RP

Calibrator: 146i 1030945145 Cal Gas: EA000737

Conc. 49.6 ppm Exp. Date 4/5/2013

NO Cal

							distance to least	acceptable	
Known Conc.	Measured Conc.	rpd	slope	intercept	rsq	forecast	squares line	distance	Pass/Fail
0	О	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

							distance to least	acceptable	
Known Conc.	Measured Conc.	rpd	slope	intercept	rsq	forecast	squares line	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

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

# NO<sub>2</sub> Calibration Spreadsheet

Station:	Rollins	Date:	5/15/2013	Operator:	JVB
Analyzer:	1125849711	Calibrator:	710902928	Cylinder:	CC08869
Cylinder Conc.	24.98	PPM			
Impurity	0.00	PPM			Yellow highlight signifies values entered b

Yellow highlight signifies values entered by user.

Green highlight signifies NO<sub>2</sub> Actual for precision data

### NO, NOx CALIBRATION

	AIR	GAS	Known	Value	Instrum	ent Response	
Calibration	Zero Air MFC	Pollutant MFC	NO	NOx	NO	NOx	NO2
	SCCM	SCCM	PPB	PPB	PPB	PPB	PPB
Zero	7993	0.00	0.0	0.0	0.0	0.0	0.0
Point 5 (50 PPB)	11976	24.02	50.0	50.0	47.5	47.8	0.2
Point 4 (100 PPB)	11952	48.04	100.0	100.0	96.4	96.7	0.3
Point 3 (200 PPB)	8928	72.06	200.0	200.0	195.7	196.4	0.7
Point 2 (300 PPB)	4920	59.76	299.8	299.8	296.3	298.2	1.8
Point 1 (400 PPB)	4920	80.06	400.0	400.0	398.3	400.3	2.0
Ozone Lamp 0%	4874	89.98					

### NO2 UPSCALE CALIBRATION (GPT)

				OI GOALL CALI	. ,					
Calibration	Inst	rument Response		C	Corrected Instrument Response and Calculated Concentrations					
points NO2	NO	NOx	NO2	[NO]ORIG	[NO]REM	[NO2]ACT				
	PPB	PPB	PPB	PPB	PPB	РРВ				
Ozone Lamp 0%	445.0	448.4	3.4	448.4	448.4	0.0				
Point 5 (50 PPB)	389.0	446.0	57	448.4	392.2	56.2				
Point 4 (100 PPB)	332.3	446.1	113.9	448.4	335.3	113.0				
Point 3 (200 PPB)	240.7	443.3	202.6	448.4	243.5	204.9				
Point 2 (300 PPB)	169.9	444.2	274.3	448.4	172.5	275.9				
Point 1 (400 PPB)	48.2	442.1	393.9	448.4	50.4	397.9				

	SLOPE	INT.
NO	0.9972	-2.0983
NOx	1.0026	-2.1821

### NO2 CONVERTER EFFICIENCY TEST

Pollutant	[NO <sub>2</sub> ]Actual	[NO <sub>x</sub> ]ORIG	[NO <sub>x</sub> ]REM	[NO <sub>2</sub> ]CONV	Converter Effiency	Is Converter Efficiency 1 ±0.04 ?
Level	PPB	PPB	PPB	PPB		
Point 5 (50 PPB)	56.2	448.4	446.0	53.8	95.7%	TRUE
Point 4 (100 PPB)	113.0	448.4	446.1	110.7	98.0%	TRUE
Point 3 (200 PPB)	204.9	448.4	443.3	199.8	97.5%	TRUE
Point 2 (300 PPB)	275.9	448.4	444.2	271.7	98.5%	TRUE
Point 1 (400 PPB)	397.9	448.4	442.1	391.6	98.4%	TRUE

Converter Efficiency (from slope)	Is Converter Efficiency 1 ±0.04 ?
98.8%	TRUE

# Form 5 – Audit Field Sheet

NO-NO <sub>2</sub> -NO <sub>x</sub> Audit Sheet	
<b>Routine Site Information</b>	

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:	
	(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 Reading:	? (Y or N)
Record NO BKG	
Record NOx BKG:	
Record PreReactor BKG:	
Record NO Coef:	
Record NO <sub>2</sub> Coef:	
Record NOx Coef:	
Reading NO:	
Reading NO <sub>2</sub> :	
Reading NOx:	
Date Last	
Zero/Prec/SpanCheckDay	/S
Ambient:	

Site:				

	0	Diagnostic Check ALARMS DETECTED:						
	Alarm	Value	<b>Corrective Action</b>					
nual	Internal Temp (°C) (15 - 45°C)		Consult Manual					
ice ma	Chamber Temp (°C) (48 – 52°C)		Check Reaction Chamber					
Make adiustments per service manua	Cooler Temp (°C) (-25 to -1°C)		Check PMT Cooler					
ustm	Conv. Temp (°C) (300 - 350°C)		Check NO <sub>2</sub> to NO Converter					
adi	Pressure (200 - 450 mm Hg)		Replace Reaction Chamber					
Make	Sample Flow (0.75 – 2.0 LPM)		Replace Pump					
	Ozonator Flow (>0.050 LPM)		Replace Pump					

 $\underline{Gas\ A\ Dilution-NO/NO_x}$ 

Zero Air	Check		Pt. 1 (0.0200499 ppm)				Pt. 2 (0.112999 ppm)			P	Pt. 3 (0.314999 ppm)				
Calibrator A	Actual:			Calibrator A	ctual:			Calibrator Actual: Calibrator			alibrator Actua	d:			
Zero Air Fl	ow:	sccms		Zero Air Flo	ow:	_sccm		Zero Air Flov	v:	sccm	Z	ero Air Flow: _	scc	m	
Gas Air Flo	ow:	sccms		Gas Air Flo	w:	_sccm		Gas Air Flow	·:	_sccm	G	as Air Flow: _	scc1	n	
	NO	$NO_2$	$NO_x$		NO	$NO_2$	NO <sub>x</sub>		NO	$NO_2$	$NO_x$		NO	$NO_2$	$NO_x$
ESC				ESC				ESC				ESC			
Reading				Reading				Reading				Reading			
% diff				% 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.														
corrected. A	An investi	gation init	nated by a	iudit results th	nat are ou	tside the a	ibove limi	ts may require	the invali	idation of	data.				

 $\frac{DataLoggerAverages(Y) - Calibrator(X)}{Calibrator(X)} \times 100 = \% \underline{\hspace{1cm}} Difference$ 

Section 3: Near-Road NO<sub>2</sub> Revised: February 17, 2016

**Revision Number: 5** 

 $GPT - NO_2$ 

 Zero Air Check
 Pt. 1 (0.020-.0499 ppm)
 Pt. 2 (0.11-.2999 ppm)
 Pt. 3 (0.31-.4999 ppm)

 GPT %:\_\_\_\_\_\_
 GPT %:\_\_\_\_\_\_
 GPT %:\_\_\_\_\_\_

Zero Air Flow: \_\_\_\_\_sccms
Gas Air Flow: \_\_\_\_sccm

	NO	NO <sub>2</sub>	NO <sub>x</sub>		NO	NO <sub>2</sub>	NO <sub>x</sub>		NO	$NO_2$	NO <sub>x</sub>		NO	NO <sub>2</sub>	NO <sub>x</sub>
ESC				ESC				ESC				ESC			
Reading				Reading				Reading				Reading			
$NO_2$				$NO_2$				$NO_2$				$NO_2$			
Actual				Actual				Actual				Actual			
% diff				% 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:

 $NO_{2Actual} = Actual \ NO_{2}$  concentration

 $NO_{2(imp)}$  = Calculated NO impurities

 $NO_{ORIG}$  = Original NO data logger reading, when  $NO_2$  is set to zero

NO<sub>REM</sub> = Remaining NO data logger reading, after addition of O<sub>3</sub>

$$NO_{2(imp)} = (\underline{F_{NO} \ (sccm))(Impurities \ from \ cylinder \ gas \ (ppb))} \\ F_{NO} + F_{O} + F_{D}$$

INUITUIT

Where:

 $F_{NO} = NO Flow$ 

 $F_0$  = Ozone Flow

 $F_D$  = Dilution Flow

# Form 6 – Audit Converter Efficiency

Impurity

# NO<sub>2</sub> Audit Spreadsheet

			- <b>L</b>		
Station:	Rollins	Date:	6/20/2013	Operator:	JVB
Analyzer:	1125849711	Calibrator:	1124449426	Cylinder:	J1363
Cylinder Conc.	24.10	PPM		•	

Yellow highlight signifies values entered by user.

Green highlight signifies NO<sub>2</sub> Actual for precision data

### NO, NOx AUDIT

	AIR	GAS	Known Value		Instrument Response		
Audit	Zero Air MFC	Pollutant MFC	NO	NOx	NO	NOx	NO2
	SCCM	SCCM	PPB	PPB	РРВ	PPB	PPB
Zero	7997	0.00	0.0	0.0	0.1	0.0	0.1
Point 4 (20-50 PPB)	15974	27.90	42.0	42.0	39.3	39.1	-0.2
Point 2 (110-300 PPB)	6947	49.38	170.1	170.1	163.9	163.9	0.0
Point 1 (310-500 PPB)	4918	82.98	399.9	399.9	390.5	390.2	-0.3
Ozone Lamp 0%	4119	78.43		<u> </u>		_	

### NO2 UPSCALE AUDIT (GPT)

PPM

		· · · · · · · · · · · · · · · · · · ·				
Audit		Instrument Resp	onse	Co	orrected Instrument Response and Cal	culated Concentrations
points NO2	NO NOx NO2		[NO]ORIG	[NO]REM	[NO2]ACT	
	PPB	PPB	PPB	PPB	РРВ	PPB
Ozone Lamp 0%	444.2	444.2	-0.1	455.4	455.4	0.0
Point 4 (20-50 PPB)	401.8	444.2	42.4	455.4	412.1	43.4
Point 2 (110-300 PPB)	295.1	443.3	148.2	455.4	303.0	152.5
Point 1 (310-500 PPB)	119.8	440.6	321.0	455.4	123.7	331.7

	SLOPE	INT.
NO	0.9780	-1.1792
NOx	0.9776	-1.2715

### NO2 CONVERTER EFFICIENCY TEST

Pollutant	[NO <sub>2</sub> ]Actual	[NO <sub>X</sub> ]ORIG	[NO <sub>X</sub> ]REM	[NO <sub>2</sub> ]CONV	Converter Effiency	Is Converter Efficiency 1 ±0.04 ?
Level	PPB	PPB	PPB	PPB		
Point 4 (20-50 PPB)	43.4	455.7	455.7	43.4	100.0%	TRUE
Point 2 (110-300 PPB)	152.5	455.7	454.8	151.5	99.4%	TRUE
Point 1 (310-500 PPB)	331.7	455.7	452.0	328.0	98.9%	TRUE

Converter Efficiency (from slope)	Is Converter Efficiency 1 ±0.04 ?
98.7%	TRUE

# Form 7 – Verification Field Sheet

NO-NO<sub>2</sub>-NO<sub>x</sub> Verification Sheet

<b>Routine Site Information</b>	
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 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)
Record NO BKG	
Record NOx BKG:	
Record PreReactor BKG:	
Record NO Coef:	
Record NO2 Coef:	
Record NOx Coef:	
Reading NO:	
<b>Reading NO2:</b>	
Reading NOX:	
Date Last	
Zero/Prec/SpanCheckDa	ays

Site:				

ALARMS DETECTED:							
Alarm	Value	<b>Corrective Action</b>					
Internal Temp (°C) (15 - 45°C)		Consult Manual					
Chamber Temp (°C) (48 – 52°C)  Cooler Temp (°C) (-25 to -1°C)  Conv. Temp (°C) (300 - 350°C)  Pressure (200 - 450 mm Hg)  Sample Flow		Check Reaction Chamber					
Cooler Temp (°C) (-25 to -1°C)		Check PMT Cooler					
Conv. Temp (°C) (300 - 350°C)		Check NO <sub>2</sub> 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					

# Gas A Dilution – NO/NO<sub>x</sub>

Ambient:\_\_\_\_\_

 Zero Air Check
 Precision Check (40-70 ppb)
 Span Check (400-450 ppb)

 Calibrator Actual:
 Calibrator Actual:

 Zero Air Flow:
 sccms
 Zero Air Flow:
 sccm

 Gas Air Flow:
 sccm
 Gas Air Flow:
 sccm

		NO	$NO_2$	$NO_x$		NO	$NO_2$	$NO_x$		NO	$NO_2$	$NO_x$
ſ	ESC				ESC				ESC			
L	reading				reading				reading			
	Drift				% diff.				% diff.			
L	Dilit				Drift				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  $\ge$ 5.0 ppb, or if the span drift is  $\pm$  10%. See Section 3.8 for calibration procedures.

Zero Drift = Current Data Logger Reading – Calibrator Reading

Revised: February 17, 2016 **Revision Number: 5** 

 $GPT - NO_2$ 

Zero Air "Orig" Check Precision Check (~50 ppb) Span Check (~400 ppb) GPT% :\_\_\_0%\_\_ GPT %:\_\_\_\_ GPT %:\_\_\_\_\_

	NO	NO <sub>2</sub>	NO <sub>x</sub>		NO	NO <sub>2</sub>	NO <sub>x</sub>		NO	NO <sub>2</sub>	NO <sub>x</sub>
ESC reading				ESC reading				ESC			
				υ				reading			
Actual NO <sub>2</sub> :				Actual NO <sub>2</sub> :				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  $\pm$  15%, or if the zero drift is  $\pm$ 5 ppb, or if the span drift is  $\pm$  10%. See Section 3.8 for calibration procedures.

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

Where:

 $NO_{2Actual} = Actual NO_{2}$  concentration

 $NO_{2(imp)}$  = Calculated NO impurities

 $NO_{orig} = Original NO conc$ , when  $NO_2$  is set to zero

NO  $_{rem}$  = Remaining NO conc, after addition of O<sub>3</sub>

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

Where:

 $F_{NO} = NO Flow$ 

 $F_0 = Ozone Flow$ 

 $F_D$  = Dilution Flow

# Form 8- Verification Converter Efficiency

Impurity

0.00

# NO<sub>2</sub> Verification Spreadsheet

Station:	Rollins	Date:	11/10/2014	Operator:	JVB
Analyzer:	1125849711	Calibrator:	710920928	Cylinder:	CB08869
Cylinder Conc.	36.10	PPM			

Yellow highlight signifies values entered by user. Green highlight signifies NO<sub>2</sub> Actual for precision data

### NO, NOx VERIFICATION

	AIR	GAS	Known Value		Instrument Response		
Verification	Zero Air MFC	Pollutant MFC	NO	NOx	NO	NOx	NO2
	SCCM	SCCM	PPB	PPB	PPB	РРВ	PPB
Zero	7998	0.00	0.0	0.0	0.0	0.2	0.2
Precision	15775	21.89	50.0	50.0	47.5	47.1	-0.4
Span (90% URL)	5917	74.84	450.9	450.9	442.9	442.8	-0.1

### NO2 UPSCALE VERIFICATION (GPT)

PPM

Verification	Instrument Response				Known Va	lues
points NO2	NO	NOx	NO2	[NO]ORIG	[NO]REM	[NO2]ACT
	PPB	PPB	PPB	PPB	PPB	РРВ
Ozone Lamp 0%	439.3	439.1	-0.3	447.3	447.3	0.0
Ozonator Mid	394.6	440.0	45.4	447.3	401.9	45.4
Ozonator High	60.5	441.7	381.2	447.3	62.3	385.0

	SLOPE	INT.
NO	0.9838	-0.8072
NOx	0.9837	-0.8867

### NO2 CONVERTER EFFICIENCY TEST

Pollutant	[NO₂]Actual	[NO <sub>x</sub> ]ORIG	[NO <sub>X</sub> ]REM	[NO <sub>2</sub> ]CONV	Converter Effiency	Is Converter Efficiency 1 ±0.04 ?
Level	PPB	PPB	PPB	PPB		
Precision	45.4	447.3	448.2	46.3	102.0%	TRUE
Span	385.0	447.3	449.9	387.7	100.7%	TRUE

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

# Form 9 – Corrective Action Form

# **Polk County Air Quality - Corrective Action Form**

To:Polk County Air Quality				
From:		(position)		
Copies of completed	form to: AQ Supervisor, File	Urgent (24 Hr.)		
Routi	ne (7 days) Next scheduled visit	Information only		
Problem Identificati	on:			
System:				
Description of Proble	m:			
Recommended Actio	n:			
Signature of Initiator:		Date:		
Problem Resolution	<b>:</b>			
Date of Corrective Ad	ction:			
Summary of Correcti	ve Action:			
Result of Corrective	Action:			
Signature of resolver	:	Date:		
Signature of OA Office	er.	Date:		