

**ELECTRONICS AND CALIBRATION BRANCH (ECB)
RESPONSIBILITIES
FOR
NITRATE MONITORING SYSTEM
RUPPRECHT AND PATASHNICK 8400N**

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**1.0 ELECTRONICS AND CALIBRATION BRANCH (ECB)
RESPONSIBILITIES
FOR
NITRATE MONITORING SYSTEM**

1.1 Overview of ECB Responsibilities

The Division of Air Quality (DAQ) of the Department of Environment and Natural Resources (DENR) operates two nitrate (NO₃) monitors, one at Rockwell (Rowan County) and another one at Millbrook (Wake County) sites in the State of North Carolina, to continuously monitor ground level ambient concentrations. The Electronics and Calibration Branch (ECB) of the Ambient Monitoring Section (AMS) of the DAQ is responsible for the selection, evaluation and procurement of the nitrate monitoring equipment and related accessories. Further, ECB is responsible for receipt, assembly, testing (at its facility) and installation of nitrate monitors in the field, evaluation of the on-going performance of nitrate monitors and related support equipment and scheduled and unscheduled system's maintenance. As a part of its responsibilities, ECB is expected to maintain a sufficient inventory of monitors, support equipment and replacement parts to minimize loss of nitrate ambient monitoring data.

Additionally, ECB staff is also responsible for procuring and maintaining dedicated traceable NO_y standard for the certification and aqueous nitrate standard for the independent accuracy auditing of the ambient air quality nitrate monitors. These trace standards provide a direct link to establish national standards and thus become basis for the collection of the highest quality ambient monitoring nitrate data possible and more so in accordance with current procedures and existing Federal Regulations and Guidelines. The continual accuracy audits performed by the ECB staff provide an ongoing evaluation of nitrate monitor's performance and Site Operator's adherence to DAQ approved operating procedures.

The ECB maintains permanent records of any standards used in the calibration and auditing of monitors and sampling equipment used in support of DAQ monitoring activities. There are permanent records at ECB for each nitrate monitors and sampler used to analyze ambient air quality in the State of North Carolina. Each major component of the nitrate monitoring system, such as Rupprecht and Patashnick (R & P) pulse generator, R & P pulse analyzer, 8400N R & P pump, etc. is assigned a dedicated logbook. These logbooks records include information related to the performance evaluations and complete records detailing the instruments and equipment placed at each monitoring site.

The ECB is also responsible for evaluating, developing and recommending changes in the equipment and operating parameters to improve the quality of data collected and procedures used in the collection of data.

1.2 Nitrate Monitoring Instrument, Equipment and Accessories

The nitrate monitoring system includes the following main components:

- R & P Pulse Generator-the main component of the nitrate monitor
- R & P Pulse Analyzer-the NO_x analyzer component of the nitrate monitor
- R & P Pump to provide vacuum to operate the system.
- Gas canisters for the nitrogen/zero purge gas supply and the NO_y in nitrogen for calibration verification.
- Commercially available aqueous nitrate standard.
- ESC Model 8832 Data Loggers, Primary (PDL). Data downloaded with the pulse generator (manually).
- A Site Dedicated PC and Modem System.

Only the main components of the nitrate monitoring system are briefly discussed here. For details of other system related components, refer to the “Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual”, by Rupprecht and Patashnick (R & P) Company, Albany, New York, 12203, February 2003.

1.2.1 8400N R & P Ambient Particulate Nitrate Analyzer

The Series 8400N R & P Ambient Particulate Nitrate Monitor measures the mass concentration of ambient particulate nitrate contained in fine particulate matter (at or below PM_{2.5}) in near real time. It measures all forms of inorganic nitrate, with no interference from ammonium salts. The Series 8400N R & P is designed to meet the United States Environmental Protection Agency (USEPA) PM_{2.5} speciation monitor requirements for the agency’s national PM chemical speciation monitoring/sampling network.

1.2.2 Principle of 8400N R & P Monitor Operation

Ambient air samples are pulled through a cyclone operated at 5.5 Lit/min to remove particles above PM_{2.5}. From this, 1 Lit/min portion of this flow is used for nitrate analysis. The 1Lit/min nitrate sample flow passes through a carbon honeycomb denuder to remove potential gaseous interferences and a Nafion humidifier to ensure that the particles are wet. Wetted ambient air particles are collected by impaction onto a nichrome strip mounted in a collection and vaporization cell. Typical sample period is 8.5 minute. After sample collection, the system switches from this collection mode to the analysis mode. During analysis step, the sample flow bypasses the collection cell, while maintaining flow through the sample line, denuder and humidifier. The collection and vaporization cell is flushed with nitrogen gas, most of which is introduced at the side of the cell (called cross-flow), but with a portion introduced through the collection orifice (called orifice flow). The nitrogen flows through the cell and into a nitrogen oxide

analyzer. The collection substrate is then flash-heated by a current from a battery until reaching an infrared cutoff. Typical heating times are 90-120 ms. Evolved nitrogen oxides are carried in the nitrogen flow to the analyzer, where they are reduced to NO by a molybdenum converter, and assayed by chemiluminescence. The analyzer output is integrated to yield the nitrate concentration. Additionally, the analyzer baseline is read prior to each analysis flash and subsequently removed from the integrated result, to yield the final, corrected sample pulse. At the end of the analysis period, the system returns to sample collection. The PM_{2.5} cyclone precut, denuder, humidifier and collection-analysis cell are housed in a box, which is ventilated with outside air to try to maintain sampling temperature close to ambient. The system outputs nitrate concentration and system operating parameters via a serial communications line at the end of each cycle.

The system may be set up to automatically conduct two types of audits: analyzer flow audits and analyzer zero/span audits. The analyzer flow audits are done during sample collection step, without interruption of the cycle. The analyzer flow audit value is used to set up the cross flow during the analysis step. Analyzer automated zero and span audits take the system off-line for two cycles. Analyzer audits may be done automatically at a preset time of the day, at a frequency of one to seven days, as selected by the Site Operator. Additionally, the system is calibrated manually using aqueous standards applied directly to the collection substrate.

For additional operating details refer to the “Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual”, by Rupprecht and Patashnick (R & P) Company, Albany, New York, 12203, February 2003.

1.3 ECB Responsibilities-Nitrate System Selection, Procurement, Receipt, Assembly and Testing

The ECB staff has responsibilities in the selection, procurement, receipt, assembly, testing (at its facility) and field installation and operation of the complete nitrate monitoring system. Since a complete nitrate monitoring system is made of various components, it is ECB responsibility to ensure compatibility of all components.

1.3.1 Nitrate Monitor System Selection and Procurement

The ECB staff will review the most recent US-EPA recommended make and model of nitrate monitor.

1.3.2 Monitor System Receipt

The nitrate monitor and its various components are shipped in number of different containers. Upon receipt of system's containers at the ECB facility, notify the carrier, if there are any damages to the containers.

At the ECB, carefully unpacked the container boxes and place each component on a stable table and leveled surface so that there is easy access to the front and back of the monitor components. Verify that all items listed on the “Receiving Report” are included. Mark each item as “Received”. Items on back order should be marked as “B. O.” and missing items should be marked as “Missing”. Sign, date and return the “Receiving Report” to the Branch Supervisor.

1.3.3 Monitor System Assembly

The ECB staff should read carefully the “Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual”, by Rupprecht and Patashnick (R & P) Company, Albany, New York, 12203, February 2003. Then only try and assemble all components of the nitrate monitoring system for testing at its facility.

In order to operate the Series 8400N R & P ambient particulate nitrate monitor, the following components needs to be set up, such as the pulse generator, pulse analyzer, pump, inlet hood, gas cylinders, in-line filters holder, computer and power cable and humidifier. Refer to above referenced operating manual, Sections 2, 3, 4 and 5 for detailed instructions for the monitor system assembly.

For the assembling of each monitor components for further testing at its facility, the ECB staff should follow all the instructions included in the “Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual”, by Rupprecht and Patashnick (R & P) Company, Albany, New York, 12203, February 2003. Specifically see Sections 2, 3, 4 and 5 of the R & P operating manual for details instructions.

1.3.4 Nitrate Monitor Testing at the ECB Facility

After assembly of the complete 8400N R & P monitor, the ECB staff will test its satisfactory operational performance at its facility. The procedural details for testing are given in the “Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual”, by Rupprecht and Patashnick (R & P) Company, Albany, New York, 12203, February 2003. Refer to above referenced manual Sections 2, 3, 4 and 5 for detailed instructions.

Specifically, after setting up the monitor, turn “on” the pulse analyzer and pulse generator and perform a primary manual leak check (R and R Operating Manual-see Section 3, sub-section 3.2.2, Page 3-19 for detailed procedure) and adjust the cell vacuum (R and R Operating Manual-see Section 3, sub-section 3.2.3, Page 3-25 for detailed procedure) before running a cycle for the first time. After turning “on” the pulse generator, allow the unit to run in “ready” mode for at least 14 hours before running the first cycle to charge the internal leas-acid battery (see Section 7 for details).

1.3.5 Inventory

Upon successful testing at its facility, ECB shall add each of monitor components to the fixed asset system. For each component namely pulse generator, pulse analyzer, etc., apply an inventory decal and make also an entry in the dedicated instrument logbook for the monitor and its site.

1.4 On-Site Installation and Testing of the Nitrate Monitoring System

In general, ECB is responsible for the installation and immediate testing, after installation (to ensure that all components of the system are operating with the prescribed limits) of all State operated monitors (including Nitrate) across the State of North Carolina. The installation of monitors at a site also includes overall satisfactory performance evaluation of nitrate monitor and all of its components. The ECB will follow US-EPA guidelines and ensure the following:

- The nitrate monitor and its associated accessories must be installed in a building where room temperature extremes do not fall below 20⁰C (68⁰F) or exceed 30⁰C (86⁰F). It shall be the Site Operator's responsibility to ensure, during each site visit that the heater and air conditioner are in working order. Remove the air conditioner filter and clean, whenever deemed necessary.
- The sample line should be 5/16" OD (outer diameter) made of stainless housed inside a 3" aspirated duct, both of which should extend approximately 2 meters above the rooftop. There should be an aluminum clamp to hold the tubing within the screened inlet hat. Make sure you have an inlet hat that is not painted.
- Install the pulse generator and pulse analyzer as per instructions in Sections 2 and 3 of the "Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual", by Rupprecht and Patashnick (R & P) Company, Albany, New York, 12203, February 2003. The pulse analyzer may be placed either alongside or underneath the pulse generator provided it is within 24 inches of the outlet located at the left-hand bottom rear of the main unit. If located underneath, it is best to construct separate shelf so that the pulse analyzer can be removed easily without disturbing the pulse generator.
- Ensure that the analyzer filter is in place prior to starting the flow to the pulse analyzer.
- Insulate the indoor portion of the 3" aspirated duct with flexible insulation such as Reflectix or fiberglass.
- With the supplied serial cable ensure that the RS-485 port on the 8400N is connected to the pulse generator with the pulse analyzer.
- Ensure that the RS-232 port on the 8400 N is connected with the supplied serial cable to a code-operated switch and then to the modem, for remote communication with the pulse generator.
- Leak test cell and inlet as per instructions in "Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual", by Rupprecht and Patashnick (R & P)

Company, Albany, New York, 12203, February 2003- see Section 3, sub-section 3.2.2, Page 3-19 for detailed procedure. Ensure that the drift is <0.01 atm/min.

- Verify system and cycle parameter settings using the table given in “Series 8400N Ambient Particulate Nitrate Monitor/Operating Manual”, by Rupprecht and Patashnick (R & P) Company, Albany, New York, 12203, February 2003. and or the Nitrate Monitor Standard Operating Procedure (SOP), January 2004.
- Perform analyzer audit as per procedure included in the Section 2.31.11.4.4.4 of the SOP.
- Start up the system and check readings as per Section 2.31.11.4 of the SOP.
- Run aqueous nitrate standards as per the procedure given in sub-section 1.6 “Accuracy Audits” of his document.
- Turn on the power to the pulse analyzer and give about 30 minutes for the ozone generator in the pulse analyzer to begin operating.
- Confirm all system parameters are set as per Table 2.32.1 of the SOP, January 2004.
- Press “RUN/STOP” to begin sampling and analysis. The 8400N is designed for automated operation and will continue sampling and analysis indefinitely barring operator intervention or component(s) malfunction.
- Pressing “RUN/STOP” again will halt sampling (with an option to abort immediately or finish the current 10 minute cycle).
- Power then can be turned off to both pulse generator and pulse analyzer.

1.4.1 Equipment Identification

ECB personnel, using Form 109 will document and log all components of the nitrate monitoring system.

1.5 8400N R & P Nitrate Monitoring System Maintenance

ECB is also intimately involved in the overall system maintenance to ensure optimum continual high data quality collection. The following are three important aspects of system maintenance that ECB is involved:

- Preventive maintenance.
- Corrective maintenance.
- Routine Maintenance.

1.5.1 Preventive Maintenance

All preventive maintenance aspects of 8400N R & P nitrate monitoring system have not been fully addressed at this time.

Note: In terms of preventive maintenance at present time, the ECB staff needs to insure that they have sufficient spare parts of the nitrate monitoring system in stock such as

carbon denuder, fan filters, etc., so that they can, upon specific request from the Site Operator, arrange to send them, on as needed basis.

1.5.2 Corrective Maintenance

All corrective maintenance aspects of 8400N R & P nitrate monitoring system have not been fully addressed by the system's manufacturer. However, it is expected that related issues will be experienced and added to this list, upon further extended field deployment of the system.

1.5.3 Routine Maintenance

As a part of routine maintenance and or during (including during site audit) any site visit, ECB will perform:

- Document the day, time and reason for the site visit in the Site Logbook.
- Check that the site building temperature is between 20⁰C and 30⁰C.
- Check that the probe and sample line are connected and secure.
- Check air conditioner, heater and lines for proper functions.
- Check that the site building is secure. Vandalism is to be reported to the ECB Supervisor.
- Check the site building for any problems (e. g. leaks, infestations, etc.).
- Check that the heat tape is working and the site insulation is adequate
- Checks that all nitrate monitoring system's components such as the pulse generator, pulse analyzer, 8400N R and P pump, etc., are operating within the prescribed ranges.
- Down any channels for nitrate monitor's component(s) repaired, replaced or audited during the repair, replacement or audit.
- Up any channels for nitrate monitor's component(s) repaired, replaced or audited during the repair, replacement or audit.
- Ensure that the "Scheduler" has been engaged before leaving the monitoring site.
- If appropriate, time duration wise change the probe every 2 years.
- Every two years, ECB staff will perform routine maintenance of the pulse analyzer (NOx) at each monitoring site using the procedures outlined in the "Electronics Calibration Branch (ECB) Responsibilities for Oxides of Nitrogen Monitoring System, Sept. 30, 2005, Rev. 5".

1.6 Accuracy Audits

During the monitoring site visit for accuracy audit, ECB staff will perform the following tasks:

- Check that the building is secured.

- Check the site temperature and make sure that it is within the set limits (20⁰C to 30⁰C).
- Check that the sample probe/sample lines are connected.
- Check that the funnel is clean and in place.
- Check that all components of the nitrate monitoring system are properly operating.
- Perform accuracy audits, in triplicate, of three different concentrations of the standard solution, using the aqueous nitrate standard solution that is commercially available. **Do not** use the nitrate standard solution that Site Operator routinely uses for system calibration. Use the following procedure:
 - On the pulse generator press “RUN/STOP” and F1 to finish current cycle or F2 to abort the current cycle.
 - Go to “Menu”-----“Service Mode”----“Aqueous Standard”.
 - Press “Start” to run one aqueous standard cycle without applying any nitrate standard solution to the strip. This step will remove any residual material from the strip and prepare it for aqueous nitrate standards.
 - Open the door of the pulse generator, locate the cell assembly and open the cell.
 - Rinse the syringe in de-ionized water, 3 times.
 - Fill the syringe to desired volume (e.g. 0.2 µl, 0.4 µl, 0.6 µl, etc.) with standard nitrate solution. Remove any air gaps in the syringe by drawing and depressing the syringe’s plunger several times while the syringe’s needle is immersed in the nitrate solution. Further, ensure that no nitrate solution drops are clinging to the outside of the needle and or syringe, by touching to mouth of nitrate standard bottle or container.
 - Apply standard nitrate solution (**Do not** use the nitrate standard solution that Site Operator routinely uses for system calibration) to the center of strip by emptying syringe and touching to strip.
 - Close the cell of the pulse generator and press, “Start” to analyze nitrate standard (the system will wait for 2 minutes and then only start sample analysis).
 - Press, “Edit” to enter the “Mass Deposit” value, in nanograms on the pulse generator screen.
 - Record the results on the “Aqueous Nitrate Standards Log” (see attached Table 1).
 - Rinse syringe thoroughly several times with de-ionized water.
 - Press “RUN/STOP” to resume normal operation.
- Calculate the percent difference, while at the site using the following equation:

$$\text{Percent Difference (\%)} = \frac{\text{Known Standard Conc.} - \text{Observed Conc.}}{\text{Known Standard Conc.}} \times 100$$

If any of the three different concentrations of nitrate audit results (in triplicate) are not within the acceptable range of $\pm 10\%$ of the expected values then do following:

- While at the site, contact the ECB Supervisor who in turn will contact the Site Operator.
- The Site Operator will flag all ambient data collected since the last acceptable nitrate standard analysis (perform such type of analysis, with at least one blank and one nitrate standard, twice a week).
- Further, the Operator will go to the site (as soon as possible), investigate any instrumental operational problems, fix the problem(s) and analyze three nitrate standards, in triplicate.
- Start collecting ambient data after ensuring that nitrate standard recoveries are within acceptable range.

As a part of the monitoring system maintenance:

- For accuracy audits ECB staff will use three different concentration of nitrate standard, each analyzed in triplicate.
- ECB will perform accuracy audits using nitrate standard solution that is different than the one that the Site Operator uses. This is to eliminate any bias.
- ECB will conduct the accuracy audit without making any instrumental changes or adjustments to the monitor.
- ECB staff will fill out the AQ 109 and AQ 121 forms, review the report and submit it to the Supervisor of the ECB within 2 workdays of conducting the audit.
- The ECB Supervisor will submit the audit report to the Section Chief of Ambient Monitoring Section within 3 workdays.

Table 1**Aqueous Nitrate Standards Log**

Site ID:
 8400N Pulse Generator Serial No.:
 Nitrate Standard Conc. (ng/ μ l):
 Nitrate Standard Solution Purchase Date@:

Date:
 Time:
 Operator:

Vol. Deposited, (μl)	Mass Deposited (ng)	Baseline (PPB)	Corrected Pulse (PPB)	Measured Mass (ng)
0.5 Blank	0			
0.5 Blank	0			
0.5 Blank	0			
0.6	60			
0.6	60			
0.6	60			
0.4	40			
0.4	40			
0.4	40			
0.2	20			
0.2	20			
0.2	20			
% Theoretical Conversion----- -----	R-Squared----- -----			

Using the data recorded, graph the “mass deposited” readings (x-axis) verses the “measured mass” readings (y-axis). Then determine the slope of the graphed line, and enter the slope in the “% of theoretical conversion”. Field in the 8400N setup screen and in the bottom line of aqueous nitrate standards log.

@Ensure that this standard is not older than 6 months (from the date of purchase).