Section 2 SOP for NO₂ May 2016 Page 1 of 47 Revision 1

FORSYTH COUNTY OFFICE OF ENVIRONMENTAL ASSISTANCE AND PROTECTION



STANDARD OPERATING PROCEDURE (SOP)

Nitrogen Dioxide (NO₂)

Section 2 SOP for NO₂ May 2016 Page 2 of 47 Revision 1

Signature Page

By the signatures below, the Forsyth County Office of Environmental Assistance and Protection (FCEAP) certifies that the information contained in the following Standard Operating Procedure (SOP) is complete and fully implemented as the official guidance for our Office. However, due to circumstances that may arise during the sampling year, some practices may change. If a change occurs, a notification of change and a request for approval will be submitted to EPA Region 4 at that time.

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Table of Contents

2.0 Introduction	5
2.1 Procurement of Calibration Standards, Zero Gases, and Monitoring Instrume	ntation5
2.1.1 General Information	5
2.1.2 Specifications for Calibration Standards, Zero Gases, and Monitoring Ins	trumentation6
2.1.3 Gas Standard and Initial Instrument Checks	7
2.2 Initial Monitor Setup	9
2.2.1 Site Requirements	9
2.2.2 Monitor Installation - Teledyne API 200EU Nitrogen Oxide Analyzer	10
2.2.3 Initial Analyzer Checks and Adjustment of the Teledyne API 200EU Nit	-
2.3 Teledyne API 200 Series Nitrogen Oxide Analyzer Instrument Description	
2.4 Multi-point Calibration of the Teledyne API 200 Series Nitrogen Oxide Anal	lyzer 16
2.4.1 Adjusted Multi-point Calibration	16
2.4.2 NO-NO _x Short Path Procedure	19
2.4.3 Ozone Presets (GPTPS)	23
2.4.4 NO-NOx GPTZ Explanation	24
2.4.5 NO2 GPT	24
2.4.15 NO/NO2/NOx 90-Day Verification	27
2.5 Teledyne API 700EU Dynamic Dilution Calibrator	28
2.6 Teledyne API 701H Zero Air Generator, Teledyne API T701H Zero Air Gen	erator28
2.7 Teledyne API 200EU Nitrogen Oxide Analyzer Maintenance	29
2.8 Routine site visits	31
2.9 Quality Assurance/Quality Control checks	32
2.9.1 Audit Short Path Procedure	32

Section 2 SOP for NO₂ May 2016 Page 4 of 47 Revision 1

2.9.2 Ozor	ne Presets fo	or Audits (GPTPS)	36
2.9.3 NO-1	NOx GPTZ	for Audits Explanation	36
2.9.4 NO2	GPT for A	udits	37
2.9.5 Bi-w	eekly Zero	/Span/Precision Checks (Z/S/P)	38
2.10 Data Ha	andling - D	ocumentation, Reduction, Analysis, and Reporting	43
REVISION	DATE	CHANGES TO SOP	
REVISION	DATE	CHANGES TO SOP	
REVISION	DATE	CHANGES TO SOP	
REVISION	DATE	CHANGES TO SOP	
REVISION	DATE	CHANGES TO SOP	
REVISION	DATE	CHANGES TO SOP	
REVISION	DATE	CHANGES TO SOP	

STANDARD OPERATING PROCEDURES NITROGEN DIOXIDE (NO₂)

Forsyth County Office of Environmental Assistance and Protection

2.0 Introduction

All equipment, chemicals, field operating procedures, and laboratory operating procedures for the continuous measurement of NO-NO_x-NO₂ in the atmosphere using the chemiluminescence method are selected and performed according to 40 CFR 50, Appendix F. The following procedure manual is to be used as a supplement to the <u>Federal Register</u> and the <u>Code of Federal Regulations</u> (CFR) for the measurement of NO-NO_x-NO₂ in the atmosphere. Siting and various quality assurance (QA) procedures are followed in accordance with <u>the EPA-454/R-98-004</u> - <u>Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II.</u>

This "Standard Operating Procedure" will provide guidance for the monitoring of NO-NO_x-NO₂ using the Teledyne API 200EU Nitrogen Oxide Analyzer (Automated Reference Method: RFNA-1194-099), Teledyne API 700EU Dynamic Dilution Calibrator and a Teledyne API T701H Zero Air Generator.

2.1 Procurement of Calibration Standards, Zero Gases, and Monitoring Instrumentation

2.1.1 General Information

- 2.1.1.1 Calibration standards include known concentrations of nitric oxides (NO/NO_x) used for calibrations, audits, precision checks, and span checks.
- 2.1.1.2 All calibration, audit, precision, and gas standards must be traceable to National Institute of Standards and Technology (NIST) Standard Reference Materials (SRM) or NIST/EPA approved commercially available certified Reference Materials (CRM); using EPA approved traceability Protocols. A "Certificate of Analysis" must accompany each gas certified to EPA Protocols. A copy of these certificates should be kept in the office by the QA staff member who oversees gas cylinder renewals. The API 700EU calibrator's Mass Flow Controllers (MFCs) must have their flow certified every 6 months and when necessary, calibrated to match a NIST traceable flow device. The flow certification process is covered in the Calibrator Operation SOP found in Section 12.
- 2.1.1.3 Zero gases are not certified to NIST standards but must meet specific requirements (see section 2.1.2.5).
- 2.1.1.4 Monitoring instrumentation must be an EPA reference or equivalent method meeting the requirements specified in 40 CFR Part 53 and 40 CFR Part 50 Appendix F.

2.1.2 Specifications for Calibration Standards, Zero Gases, and Monitoring Instrumentation

2.1.2.1 Calibration gases will contain NO in nitrogen in the range of 6 - 60 ppm. Gases will be analyzed for NO and NO_x and certified as described in section 2.1.1.2. Cylinder gases will be diluted to the appropriate concentrations using a dynamic gas dilution system incorporating gas phase titration with ozone for the production of NO₂. See Figure 1.



Figure 1: Certificate of Analysis for Gas Cylinders

2.1.2.2 NO/NOx and NO₂ concentrations used for multi-point calibrations are produced by a gas dilution system and should be in the following ranges:

Point 1: 0 ppb NO/NOx (Zero) Point 1: 0 ppb NO₂ (Zero)

Point 2: 225 ppb NO/NOx (Span) Point 2: 190 ppb NO₂ (Span)

Point 3: 160 ppb NO/NOx (Midpoint) Point 3: 160 ppb NO₂ (Midpoint)

Point 4: 70 ppb NO/NOx (Precision) Point 4: 70 ppb NO₂ (Precision)

Point 5: 40 ppb NO/NOx (Low-point) Point 5: 40 ppb NO₂ (Low-point)

2.1.2.3 Audit concentrations must be produced by a system independent of the routine calibration system. A minimum of zero and three upscale points should be chosen to bracket 80% of the ambient data if at all possible. The points chosen must be in the following ranges, contained within the

Section 2 SOP for NO₂ May 2016 Page 7 of 47 Revision 1

calibration range that the FCEAP uses, which is 0-250 ppb. For FCEAP, three of the points chosen must be in the required levels in the following ranges. Additional points can be added and run in any other level.

Level 1: 0.3-2.9 ppb NO₂ (Required)

Level 2: 3.0-4.9 ppb NO₂

Level 3: 5.0-7.9 ppb NO₂

Level 4: 8.0-19.9 ppb NO₂

Level 5: 20.0-49.9 ppb NO₂ (Required)

Level 6: 50.0-99.9 ppb NO₂

Level 7: 100.0-299.9 ppb NO₂ (Required)

Level 8: 300.0-499.9 ppb NO₂ (Over FCEAP range)

Level 9: 500.0-799.9 ppb NO₂ (Over FCEAP range)

Level 10: 800.0-1000.0 ppb NO₂ (Over FCEAP range)

Audit standards must be independent of the standards used for calibrations/verifications.

2.1.2.4 NO₂ concentrations used to perform zero/span/precision checks are produced by a gas dilution system and must be in the range of:

Zero: 0.0 ppb NO₂

Span: 170 - 250 ppb NO₂ Precision: 60 - 80 ppb NO₂

2.1.2.5 Zero air to be used for calibrations, 90-day verifications, bi-weekly Zero/Span/Precision (ZSP) checks, and audits must be free of contaminates, which will cause a detectable response on the NO2 analyzer. The zero air should contain < 1.0 ppb of NO2. A series of drierite columns or similar containers loaded with purafil, silica gel, charcoal, hopcalite, and molecular sieve is used to scrub compressed air. The compressed air is routed through a 5 μ m Teflon filter.

Audit zero air is provided by a pump (diaphragm or oil-less piston) moving air through a series of scrubbers. The audit zero air is dried with silica gel, then scrubbed through purafil and charcoal. The audit zero air is finally filtered through a $5 \mu m$ particulate filter.

2.1.3 Gas Standard and Initial Instrument Checks

- 2.1.3.1 Upon receipt of gases, check to insure that the certificate of analysis is included with each cylinder.
- 2.1.3.2 Check the concentration on the cylinder label against the concentration on the certificate for each cylinder.
- 2.1.3.3 Thoroughly check each gas cylinder to ensure that all specifications have been met by running a ZSP check on an up to date, calibrated, analyzer. Reject any gases that do not pass specifications and return them to the supplier.

- 2.1.3.4 Upon receipt of cylinder gas standards the following information must be clearly marked on the cylinder by affixing a tag to the cylinder:
 - a: ID Reference Number
 - b: Cylinder contents
 - c: Cylinder concentrations
 - d: Expiration date
 - e: Cylinder usage (i.e. cal, span, precision, etc.)

Cylinder standards must not be used after the expiration date until recertified.

- 2.1.3.5 NO2 instrumentation must meet the requirements of the Technical Assistance Document for Precursor Gas Measurements (EPA -454/R-05-003, September 2005) or be an equivalent method as described in 40 CFR, Part 53. A list of EPA designated reference and equivalent methods is available from EPA.
 - a: An EPA designation sticker must be affixed to the instrument.
 - b: A factory manual must accompany the instrument.
 - c: A record or log (hardcopy) of all maintenance done to the analyzer must be kept in the pocket on top of the analyzer. Update digital copy on shared drive as well.
 - d: Instrument must be tested and performance documented in the FCEAP master Excel spreadsheet (Fig. 2) containing all check records for network equipment. This document is located in the S:\A&M\Repair Supplies and Logs\Instruments checks folder and is called 200EU T200U Analyzer In-Lab checks.xls. Below is the layout:

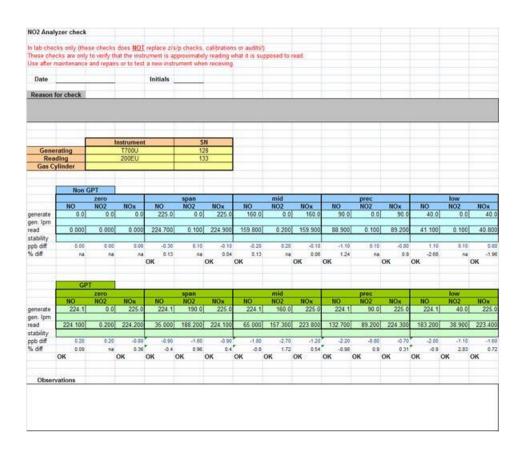


Figure 2: Instrument Checks Form

After calibration, if 2% error tolerances are not met, inform the Program Manager and contact the manufacturer.

2.2 Initial Monitor Setup

2.2.1 Site Requirements

To ensure the uniform collection of air quality data, various siting criteria must be followed. 40 CFR 58 Appendix E outline these criteria. The criteria are summarized below for middle, neighborhood, and urban spatial scales. Our NO2 is run on the neighborhood scale.

- 2.2.1.1 The sample probe inlet must be located 2-15 m above ground and a distance from the supporting structure >1 m.
- 2.2.1.2 The probe inlet should be >10 m from the drip line of trees that are located between the urban city core and along the predominant summer daytime wind direction.

- 2.2.1.3 The distance from the probe inlet to any obstacles such as buildings must be at least twice the height the obstacle protrudes above the probe inlet.
- 2.2.1.4 There must be unrestricted airflow 270° around the inlet probe, or 180° if the probe is on the side of a building. The 270° arc must include the predominant wind direction for the season of greatest pollutant concentration. In the Winston-Salem, NC area the primary wind direction is SW.
- 2.2.1.5 The sample line should be as short as practical and should be constructed of borosilicate glass, FEP Teflon, or their equivalent.
- 2.2.1.6 If the above siting criteria cannot be followed, it must be thoroughly documented and a waiver requested from the Air, Pesticides and Toxics Management Division (APTMD) of the USEPA. A complete site evaluation including all dimensions, pictures, maps, and the monitoring objective should be prepared as the site is being set up. This documentation should be maintained in the annual monitoring network plan.

2.2.2 Monitor Installation - Teledyne API 200EU Nitrogen Oxide Analyzer

- 2.2.2.1 The analyzer should be placed on a sturdy table or in an appropriately sized instrument rack.
- 2.2.2.2 The table or rack should be as vibration free as possible.
- 2.2.2.3 The analyzer must operate within the temperature range of 20 30 C.
- 2.2.2.4 A verified thermometer should be installed near the analyzer to observe temperature readings to insure that temperature criteria are met. It is polled and checked along with other data to make sure it falls within limits. Identify and correct problem if it is not within limits. The thermometer will be verified semi-annually to ensure proper function (Fig 3).



Figure 3: Site temperature device

2.2.2.5 Connect ambient air to be measured to the bulkhead connector labeled "SAMPLE" on the rear panel of the instrument. Care should be taken to see that dirty, wet, or incompatible materials in the sample lines do not contaminate the sample. Teflon tubing with an OD of 1/4" and a minimum ID of 1/8" is required for all sample lines. The length of the tubing should be held to a minimum. Connect the rear panel bulkhead labeled 'Exhaust' to a suitable charcoal scrubber. The exhaust stream will

Section 2 SOP for NO₂ May 2016 Page 11 of 47 Revision 1

contain significant concentrations of ozone and oxides of nitrogen. The exhaust should be vented to the outside of the building.

- 2.2.2.6 Confirm that an 5-µm Teflon particulate filter is installed in the filter holder and the holder is connected to the sample line before the sample port.
- 2.2.2.7 Plug in analyzer.
- 2.2.2.8 Turn on power switch.
- 2.2.2.9 Check that the instrument is booting the firmware. Let it warm up for at least 1 hour. Use the menu on the front panel to check the instruments diagnostics. If the sample flow is outside its ranges, check for blockages, pump condition, and/or leaks.
- 2.2.2.10 Connect the calibration standard gas produced from the calibrator to the NO/NO2 solenoid (Fig 4) valve that feeds NO/NO2 calibrator concentrations to the sample probe box outside the building via a Teflon (FEP) line with OD of 1/4" and a minimum ID of 1/8". The transfer standard will send NO/NO2 concentrations up the cal gas line into the probe box to a "tee". The tee is also connected to the short inlet line that goes to the inlet funnel and the sample feed going to the analyzer. In ambient operation the analyzer pulls ambient air from the inlet line and the cal gas line is sealed by the solenoid. In calibration operation the transfer standard supplies NO/NO2 concentrations through the solenoid and cal gas line up to the probe box. The analyzer pulls what it needs through the sample line and the inlet line becomes the vent for the excess cal feed. The entire sample path except the short inlet line (less than 12") is used during all reportable QC/QA checks.

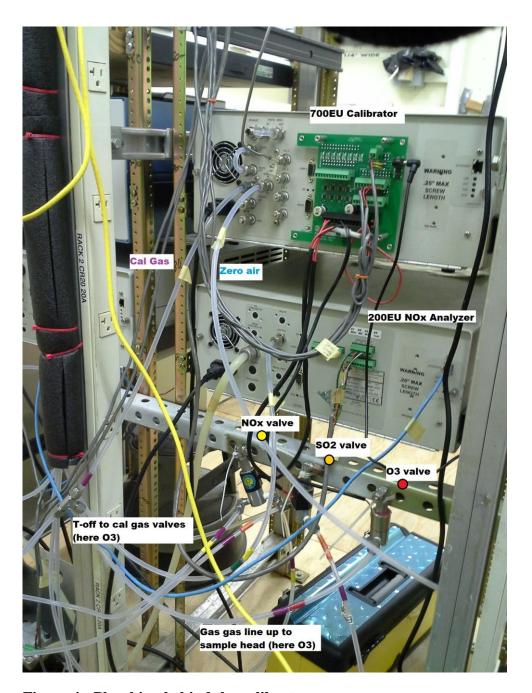


Figure 4: Plumbing behind the calibrator

- 2.2.2.11 An ESC 8832 Datalogger is used as the Datalogger. The TAPI NO2 Analyzer is connected to the 8832 through an analog connection. Configurations for individual channels are programmed into the 8832 Datalogger, site computer, and Office polling computer. Refer to Section 11 Datalogger 8832 SOP for more information. Check that the Datalogger channel has been properly initialized as follows:
- 2.2.2.11.1 To Login into the 8832, open 'HyperTerminal' on the PC and connect to the 8832 through the appropriate COM port or by using the correct IP address.

Type Esc 'site ID' AQM to enter Login screen.

Site ID in the Forsyth County Network:

Clemmons Middle (CM)

Hattie Avenue A (HA)

Hattie Avenue B (HB)

Union Cross (UC)

Press L (Login), type password, press Enter. Then press C (Configuration Menu), D (Configure (Data) Channels), C (Change Old Configuration). Press Enter to see the channel configurations.

2.2.2.11.2 Check the channel configuration entries (Fig. 5a/5b/5c) to ensure that they correspond to the entries listed below:







Figure 5a/5b/5c: 8832 NO/NO2/NOx channel configuration

2.2.2.12 The internal memory on the analyzer is used a back up data logger. The site computer utilizing E-DAS Digi-Trend software is also used to backup the hourly data from the Datalogger. The local computer polls the minute and calibration data from the 8832 Datalogger each hour to maintain a local copy on site. Using the slope and intercept from the 200EU/T200U records calculation we can recover lost hourly data from the minute data on the 8832.

2.2.3 Initial Analyzer Checks and Adjustment of the Teledyne API 200EU Nitrogen Oxide Analyzer

- 2.2.3.1 Turn the instrument's power switch to ON. The instrument will display various 'Loading' screens while it is warming up and conducting self-tests. After loading has finished, press 'Clr' button on the instrument to clear the 'System Reset' warning message.
- 2.2.3.2 To set the time and date press 'Setup, Clk, Time (EST) or Date' on the instrument and adjust using the corresponding buttons, press 'Enter' to save your settings and 'Exit' back out to the main screen. To set the ambient pressure, go into the 'Diagnostics' menu press 'Next' button to get to PRES in-Hg-A and press 'Enter', set the pressure and press 'Enter' to save.
- 2.2.3.3 Allow the instrument to warm up for at least 1 hour.
- 2.2.3.4 Using the 'Test' button on the front panel (Fig 6), check the following diagnostics:



Figure 6: Front panel 200EU NOx Analyzer

Sample flow $1000 \pm 50 \text{ cm}^3$ (> 700 cm^3) Ozone flow $80 \pm 10 \text{ cm}^3$ RC temp $40 \pm 1 ^{\circ}\text{C}$ Box temp $8 - 48 ^{\circ}\text{C}$ PMT temp $5 \pm 1 ^{\circ}\text{C}$ MF temp $8 - 48 ^{\circ}\text{C}$ CNV temp $315 \pm 5 ^{\circ}\text{C}$ RC press < 10 in-Hg-ASM press 25 - 34 in-Hg-A (~1" < Ambient)

If any of these diagnostic values are not met, perform troubleshooting and necessary repairs/maintenance (see chapter 2.7).

- 2.2.3.5 Check the HVPS value on the front display (use the 'Test' buttons). The HVPS has to be between 450-900V. If it is outside this range, a PMT Adjustment is necessary, refer to Teledyne API Service Note 13-002 'Performing a PMT Adjustment on a NO/NO_x Analyzer'.
- 2.2.3.6 Calculate the Moly Converter Efficiency. To ensure accurate operation of the 200EU Nitrogen Oxide Analyzer, it is important to check the NO₂ converter efficiency (CE) during 90-day verification checks. For the analyzer to function correctly, the CE must be between 0.960 and 1.020 (96-102%). If the CE is outside these limits, the NO₂ converter should be replaced. Refer to Teledyne API Service Note 04-001 RevC (17-May-2010) "How to calculate moly converter efficiencies."

A prepared table is in the instrument log book (see Fig. 7).

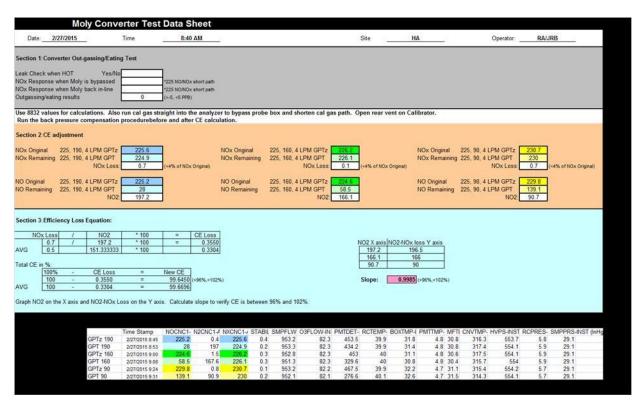


Figure 7: Converter Efficiency Calculation worksheet

2.3 Teledyne API 200 Series Nitrogen Oxide Analyzer Instrument Description

The 200EU instrument is a microprocessor controlled instrument that determines the concentration of NO, total NO_x (sum of NO and NO_2) and NO_2 in a sample gas drawn through the instrument.

It requires that sample and calibration gases be supplied at ambient pressure in order to establish a constant gas flow through the reaction cell where the sample gas is exposed to O_3 , where one NO molecule will chemically react with one O_3 molecule, producing O_3 and excited NO*₂. The excited

Section 2 SOP for NO₂ May 2016 Page 16 of 47 Revision 1

NO*₂ condition is unstable, forcing it to release energy to return to a stable NO₂ state, hereby giving off a quantum of light (hv) with a peak at 1200nm.

The instrument measures the amount of chemiluminescence to determine the amount of NO in the sample gas. A catalytic-reactive converter converts NO_2 in the sample gas to NO which, along with the NO present in the sample gas, is reported as NO_x . The NO_2 is calculated as the difference between NO_x and NO.

2.4 Multi-point Calibration of the Teledyne API 200 Series Nitrogen Oxide Analyzer

NO-NO₂-NO_x analyzers are to be calibrated upon receipt, when installed, if moved from current location, and when certain repairs are made. An adjusted calibration may be necessary if an analyzer malfunctions and is repaired, or if power is lost for more than 24 continuous hours at a site.

Before the actual initial calibration is performed, the moly converter efficiency should be calculated. See section 2.2.3.6. This ensures accurate operation of the 200EU Nitrogen Oxide Analyzer.

An Adjusted Calibration, during which the lowest point (Zero) and the highest point (Span) are adjusted on the analyzer itself, is used at the start of sample collections for a site, and/or when a biweekly ZSP check or 90 day verification fails; but operator must contact Program Manager before proceeding directly to a calibration if QC checks fail. The resulting slope and intercept values are automatically stored in the instruments memory. In addition, a new slope and intercept will be calculated comparing the analog voltage (from the instrument) and the engineering units output from the 8832 datalogger in the Excel site logbook. This updated slope and intercept will be entered into the 8832. The adjusted calibration resets the performance check (Bi-weekly Zero/Span/Precision) schedule, starting with the performance date of the Adjusted Calibration.

During a 90 day verification (multipoint check - 4 points plus a zero) the results are recorded in "as found" condition. The 90 day verification does not reset the Bi-weekly Zero/Span/Precision (ZSP) schedule. The (ZSP) will remain on its previous schedule.

2.4.1 Adjusted Multi-point Calibration

2.4.1.1 Typically the only time a Calibration is performed will be at the beginning of the analyzer's field operation or after certain maintenance or repairs.

A calibration must be performed if a 90-day verification or bi-weekly zero/span/precision (ZSP) check fails and the instrument is in good working order. Normally if either of these checks fail there is some problem within the monitoring system that needs addressing. If the Zero check is outside >± 0.005 ppm of known 0.000 or the Span check >± 10% of expected value, then an adjusted calibration will be done AFTER equipment failure is diagnosed, repaired, and instrument cleared for normal operation. If a typical slow drift causes the check to fail, no maintenance may be necessary but check with the program manager before proceeding.

Section 2 SOP for NO₂ May 2016 Page 17 of 47 Revision 1

- 2.4.1.2 Allow sufficient time for the NO₂ analyzer and the calibration standard to warm up (~1 hour) as necessary, if they are not already on.
- 2.4.1.3 Always, if no major malfunctions have occurred and the monitor has been in normal operation, perform a bi-weekly zero/span/precision (ZSP) check prior to a calibration. If necessary, after the ZSP check, install a clean 5-µm particulate filter in the monitor filter holder in the probe line box on the roof of the site. Perform a system leak check (refer to 43i-tle manual) after replacing the filter and saturate the probe system with NO-NO_x by running a NO-NO_x span point (225 ppb) for 15 minutes. Record all information in the logbook.
- 2.4.1.4 An Adjusted Calibration procedure consists of four major steps:
 - Short Path (no O₃) (2.4.2)
 - Ozone Presets (on the Calibrator) (2.4.3)
 - Gas Phase Titration (GPT; O₃ introduced) (2.4.5)
 - ESC 8832 data logger Update (2.4.2 & 2.4.5)
- 2.4.1.5 Login into the ESC 8832 data logger using 'HyperTerminal' on the PC.

Refer to Section 11 Datalogger 8832 SOP for more information.

Type Esc (two letter site ID) AQM Enter, to enter Login screen.

ESC 8832 v3.02 ID:HA

Home Menu

11/06/14 14:01:50

H Help Screen

L Login / Set User Level

O Log Out / Exit

Press L (Login), type password, press Enter. C (Configuration Menu), D (Configure (Data) Channels), C (Change Old Configuration). Press M (Disable/Mark Channel Offline). Use arrows to skip to NO-NO2-NOx, then press Enter for each to disable the all three channels.

2.4.1.6 Prepare a calibration worksheet in the instrument logbook containing the following entries:

Date/Time; Operator; Site/AQS ID; Datalogger check; Analyzer and Calibrator Make/Model/Serial Number/Diagnostics; NO-NO2-NOx, Operational checks and NO-NO2-NOx, Readings

The following example of the electronic data sheet (Fig. 8) will be used to document checks. All information fields listed above must be included.

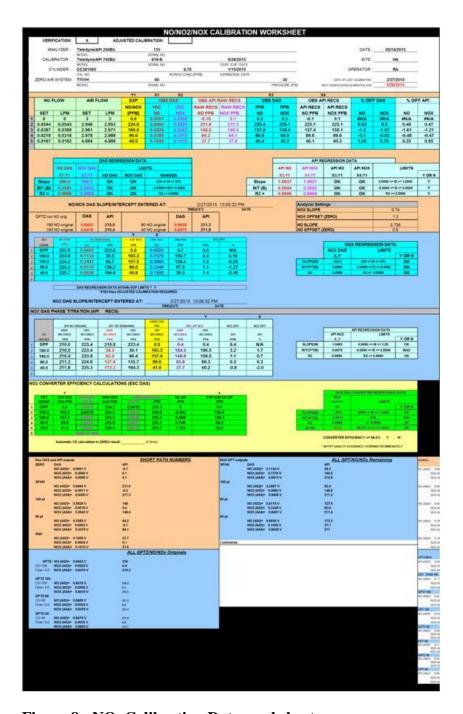


Figure 8: NO₂ Calibration Data worksheet

- 2.4.1.7 In the ESC 8832 skip back to the Main Menu (use Esc). Press D (Real-Time Display), V (Display Raw Readings).
- 2.4.1.8 Make sure the 700EU Calibrator is connected to a source of zero air (i.e. Teledyne API T701H) which is reading a pressure of 25-30 psig. Check the regulator pressure on the 700EU Calibrator to make sure it is at 7-10 psig. Check a source of calibration gas (Gas cylinder) is connected to the 700EU Calibrator with a pressure of 20-25 psig.

- 2.4.1.9 Check that the 700EU Calibrator 'Cal Gas Out' is connected to the 200EU Nitrogen Oxide Analyzer 'Sample Inlet'. Make sure the flow of calibration gas is routed through the analyzer particulate filter. The test atmosphere must pass through all filters, conditioners, other components used during normal ambient sampling, and as much of the ambient air inlet system as is practicable.
- 2.4.1.10 Calculate the moly converter efficiency (see chapter 2.2.3.5).

2.4.2 NO-NO_x Short Path Procedure

During the Short Path part of the calibration, no ozone is introduced and only the NO-NO_x values are recorded. The only NO₂ value used is the zero point NO₂ observed 8832 and 200EU reading.

2.4.2.1 On the 700EU calibrator press SEQ (Fig 9), use the arrow keys to reach NO_x ZERO, press Enter to start the sequence.



Figure 9: Calibrator Sequence Program

- 2.4.2.2 Check the instrument functions by pressing the Test button. Check the analyzer temperatures, pressure, flow, and intensities. Any issues shall be addressed before completing the calibration using the manufacturer's manual.
- 2.4.2.3 Allow the analyzer to sample zero air for at least 30 minutes until stability < 0.2 is obtained. If the analyzer needs to be adjusted to read zero, press the CAL button and choose ZERO, press Enter. Return to the main screen (press Exit) and let the analyzer stabilize. The analyzer should now read zero, if not inform the Program Manager.

<u>It is recommended to wait for a very low stability and calibrate the point once instead of calibrating the point consecutively until the point becomes stable. Check the Digitrend charts to assist with this.</u>

2.4.2.4 Allow the instrument to stabilize (~30 minutes). Observe the analyzer and the calibration standard. The difference for the zero point should meet the following specification:

2.4.2.5 Record (highlight, right click 'copy') the raw voltage reading (Fig. 10) from the ESC 8832 (in Hyperterminal) and download the instrument's 1-minute readings using the APICom software. Open APICom 4.0.1exe, select the 200EU and click the 'green arrow' to open a 200EU front display window (Fig 11a).

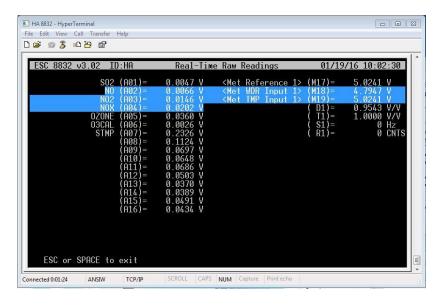


Figure 10: ESC 8832 Raw Voltage Reading



Figure 11a: APICom Home

Click the 'iDAS' button (Fig 11b).



Figure 11b: APICom Analyzer Screen

In the opening window, mark the NONOX_MIN box, click the 'Get Data' button, choose 'most recent on record' to download the 1-minute readings from the 200EU. Click 'Save Data' (to 200EU NOx mindata-test.vars.csv file) and choose 'Append' when asked (Fig 11c).

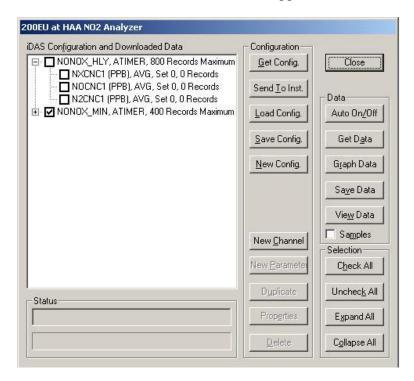


Figure 11c: APICom iDAS Screen

Paste both, the data logger (ESC) and copy/paste APICom min data, readings into the instruments logbook (Fig. 8). The following observed DAS and API NO and NO_x readings have to be manually copied into OBS DAS VDC NO; DAS VDC NO_x and OBS API Raw Recs NO; OBS API Raw Recs NO_x cells (see Appendix: 'NOx Calibration worksheet' for assistance).

2.4.2.6 While the zero point is still running, on the 700EU Calibrator check the NO Flow and Air Flow settings and actual flows. Copy these readings to the NO FLOW Set/Lpm and AIR FLOW Set/Lpm in the worksheet. Check the expected NO/NO_x values and copy it to the EXP NO/NOx [PPB] in the worksheet.

- 2.4.2.7 Press SEQ on the 700EU Calibrator, use the arrow keys to reach NO_x225 , press Enter to start the Span point.
- 2.4.2.8 Perform the following steps until no further adjustments are necessary. Record results after all adjustments are complete.
- 2.4.2.9 Allow the analyzer to sample 225 ppb (Span) gas for about 15 minutes until stability < 0.2 is obtained. If the analyzer needs to be adjusted to read 225 ppb, press the CAL button and choose SPAN, press Enter. Return to the main screen (press Exit) and let the analyzer stabilize. The analyzer should now read 225 ppb, if not repeat the adjustment steps above.

<u>It is recommended to wait for a very low stability and calibrate the point once instead of calibrating the point consecutively until the point becomes stable. Check the Digitrend charts to assist with this.</u>

2.4.2.10 Allow the instrument to stabilize (15-30 minutes). Observe the analyzer and the calibration standard. The difference for the span point should meet the following specification:

 $\leq \pm 2$ ppb at stability of < 0.5

- 2.4.2.11 Record the raw voltage reading from the ESC 8832 and download the instrument's 1-minute reading using APICom (see 2.4.2.5). Paste (ESC) and copy/paste (APICom) both readings into the instruments logbook (Fig. 8). The following observed DAS and API NO and NO $_x$ readings have to be manually copied into OBS DAS VDC NO; DAS VDC NO $_x$ and OBS API Raw Recs NO; API Raw Recs NO $_x$ cells (see Appendix: 'NO $_x$ Calibration worksheet' for assistance).
- 2.4.2.12 While the span point is still running, on the 700EU Calibrator check the NO FLOW Set/Lpm and AIR FLOW Set/Lpm settings. Check the EXP NO/NO_x [PPM] values.
- 2.4.2.13 After the 0.0 ppb (Zero) and 225 ppb (Span) NO-NO_x points have been run satisfactorily and data recorded, start the sequences on the 700EU Calibrator to run the points for NO_x 160 ppb, NO_x 90 ppb, and NO_x 40 ppb. Press SEQ on the 700EU Calibrator, use arrow keys to reach the desired NO-NO_x concentration, press Enter.

Do NOT adjust the analyzer while running any of the midpoints.

Record the results for each concentration based on stable readings (stability <0.5) from the ESC 8832 Raw Readings and 1-minute APICom (see 2.4.2.5) in the instrument logbook (Fig. 8) and manually copy them into the corresponding OBS DAS VDC NO; OBS DAS VDC NO_x and OBS API Raw Recs NO; OBS API Raw Recs NO; Cells. The difference for each point should meet the following specification:

 $\leq \pm 2\%$ difference

If it is not within the % difference for each point inform the Program Manager.

Check the NO FLOW Set/Lpm and AIR FLOW Set/Lpm settings. Check the EXP NO/NO_x [PPM] values.

2.4.2.14 Review the linear regression results for NO/NOx calculated in the calibration worksheet between the expected NO/NOx and the observed NO/NOx from the 8832. The linear regression line should meet the following specifications in order to be valid for reporting ambient air data: $245 \le m \le 255$, $-0.500 \le b \le 0.500$ and $r2 \ge 0.9990$ (the logger slope and intercept translates the raw voltage into engineering units for the data logger). If the line does not meet these specifications inform the Program Manager. If specifications are met, enter the new slope and intercept into the NO/NOx channel configuration in the 8832.

2.4.2.15 Review the linear regression results for NO/NOx calculated in the calibration worksheet between the expected NO/NOx and the observed NO/NOx from the 200EU. The linear regression line should meet the following specifications in order to be valid for reducing ambient air data: 0.9800 < slope < 1.0200, -2.0 < intercept < 2.0, and $r \ge 0.9990$ (the analyzer slope and intercept adjusts a ppb value to a corrected ppb value based on a best fit line across the five points). If the line does not meet these specifications inform the Program Manager. If specifications are met, the new slope and intercept can be applied to any value stored in the internal datalogger in the analyzer if data is lost from the 8832.

If these specifications are not met, corrective action should be taken and another calibration should be performed after problem is identified and corrected.

2.4.3 Ozone Presets (GPTPS)

2.4.3.1 Before continuing the calibration procedure for NO2 with the Gas Phase Titration (GPT) part, presets have to be run on the 700EU Calibrator. Press SEQ on the 700EU Calibrator, use arrow keys to reach EXE.SEQ.PS, press Enter.

The preset mimics the 700EU Calibrator set up for running the following GPT without mixing any O3 with calibration gas. Instead, the internal photometer measures the actual ozone concentration and adjusts the ozone drive voltage on the ozone generator, to receive a most accurate NO₂ reading later during the GPT.

The preset will run approximately 15-20 min and no records have to be taken during the preset, as this only prepares the 700EU Calibrator for the following GPT. Observe the 'Active' and 'Auto' lights on the front panel (Fig 12).



Figure 12: 700EU Active/Auto lights

Section 2 SOP for NO₂ May 2016 Page 24 of 47 Revision 1

While both lights are blinking the calibrator is adjusting the ozone drive voltage. When the 'Active' light is steady lit, it's setting a reference point. There will be a total of 8 steps to complete the presets sequence.

Once the 700EU Calibrator is back in Standby mode, proceed with the Gas Phase Titration Zero (GPTZ) procedure.

2.4.4 NO-NOx GPTZ Explanation

During the GPTZ, ozone is not introduced to the calibration gas mixture but the flow paths and amounts follow the GPT settings for a given desired result. The GPTZ steps will produce the NO_{orig} and NOx_{orig} (original) values used along with NO_{rem} and NOx_{rem} (remaining) collected during the GPT steps to calculate expected NO2 levels.

- 2.4.4.1 On the calibrator press SEQ, use the arrows to reach GPTZ, press Enter. This will start the zero point for the GPT run. This GPTZ point will mimic the flow settings for the 190 ppb NO2 GPT point but will serve as the zero point for NO2. During the GPTZ 160, GPTZ 90 and GPTZ 40 the NO_{Orig} and NO_{xOrig} are obtained to get more accurate NO_{Rem} and NO_{2Rem} calculations. On the 700EU Calibrator press SEQ, use the arrow keys to reach the desired GPTZ point. The results can be used as the NO_{orig} and NOx_{orig}.
- 2.4.4.2 Allow the instrument to stabilize (15-30 minutes). Observe the analyzer and the calibration standard. The % error for the GPTZ points should meet the following specification:

 $\leq \pm 10\%$ ppb of 225ppb at stability of < 0.5

*Note: Each GPT point that will be run will have a GPTZ point ran right before it with the same flow and desired ozone level targets as the GPT.

No adjustments of the NO-NO_x-NO₂ values during the entire GPTZ or GPT phases are necessary!

2.4.4.3 Copy the raw voltage readings from the ESC 8832 and download the instrument's 1-minute reading using APICom (see 2.4.2.5). Copy both readings into the instrument's logbook (Fig. 8). The following observed DAS and API NO, NO_x and NO₂ readings have to be manually copied into DAS NO_{Rem} VDC; DAS NO_{2Obs} VDC; DAS NO_{xOrig} Volts and API NO_{Orig} Raw Records; API NO_{2Obs} Raw Records cells. Also enter the NO_{Orig} DAS and API in 'GPTZ run NO orig / 190 NO original' (see Appendix: 'NOx Calibration worksheet' for assistance).

2.4.5 NO2 GPT

- 2.4.5.1 On the 700EU Calibrator press SEQ, use the arrows to reach NO2 190 (Span point), press Enter. This step actually mixed ozone into the NO/NOx gas to produce a given NO2 point.
- 2.4.5.2 Allow the instrument to stabilize (15-30 minutes). Observe the analyzer and the calibration standard. The difference for all NO2 points should meet the following specification:

Section 2 SOP for NO₂ May 2016 Page 25 of 47 Revision 1

 $\leq \pm 10\%$ difference of the calculated expected ppb for NO2 at stability of < 0.5

- 2.4.5.3 Copy the raw voltage reading from the ESC 8832 and download the instrument's 1-minute reading using APICom (see 2.4.2.5). Copy both readings into the instrument's logbook (Fig. 8). The following observed DAS and API NO, NO_x and NO₂ readings have to be manually copied into DAS NO_{Rem} VDC; DAS NO_{2Obs} VDC; DAS NO_{xRem} Volts and API NO_{Rem} Raw Records; API NO_{2Obs} Raw Records cells (see Appendix: 'NOx Calibration worksheet' for assistance).
- 2.4.5.4 After the 0.0 ppb (Zero) and 190 ppb (Span) GPT points have been run satisfactorily and recorded, start the sequences on the 700EU Calibrator to run points for GPT NO₂ 160, GPT NO₂ 90 ppb and GPT NO₂ 40 ppb.
- *Before each of the following GPT points are to be run, a GPTZ point has to be run with the same target ozone and total flow to be used for each of the GPT points. See 2.4.4.
- 2.4.5.5 After the GPTZ point, press SEQ on the 700EU calibrator, use arrow keys to reach the desired GPT NO₂ concentration, press Enter.

Record the results for each concentration based on stable readings on the analyzer (stability <0.5) and using the digitrend chart. Copy the raw voltage readings from the ESC 8832 and the instrument's 1-minute reading using APICom (see 2.4.2.5) in the instrument logbook (Fig. 8) and manually copy into the corresponding into DAS NO_{Rem} VDC; DAS NO_{2Obs} VDC; DAS NO_{xOrig} Volts; DAS NO_{xRem} Volts and API NO_{Rem} raw records; API NO_{2Obs} raw records cells. From each associated GPTZ manually copy the API NO_{Orig} and DAS NO_{xOrig} VDC values (see Appendix: 'NOx Calibration worksheet' for assistance).

- 2.4.5.6 Review the linear regression results for NO2 calculated in the calibration worksheet between the expected NO2 and the observed NO2 from the 8832. The linear regression line should meet the following specifications in order to be valid for reporting ambient air data: $245 \le m \le 255$, $-2.0 \le b \le 2.0$, and $r2 \ge 0.9990$ (the logger slope and intercept translates the raw voltage into engineering units for the data logger). If the line does not meet these specifications inform the Program Manager. If specifications are met, enter the new slope and intercept into the NO2 channel configuration in the 8832.
- 2.4.5.7 Review the linear regression results for NO2 calculated in the calibration worksheet between the expected NO2 and the observed NO2 from the 200EU. The linear regression line should meet the following specifications in order to be valid for reducing ambient air data: 0.9800 < slope < 1.0200, -2.0 < intercept < 2.0, and $r \ge 0.9990$ (the analyzer slope and intercept adjusts a ppb value to a corrected ppb value based on a best fit line across the five points). If the line does not meet these specifications inform the Program Manager. If specifications are met, the new slope and intercept can be applied to any value stored in the internal datalogger in the analyzer if data is lost from the 8832.

If not, inform the Program Manager.

- 2.4.5.8 Close all APICom windows to disconnect from the NO2 Analyzer.
- 2.4.5.9 On the 700EU Calibrator press the STBY button to bring it back in standby mode. Check the analyzer for it to return to reading ambient NO-NO_x-NO₂ values.

- 2.4.5.10 The newly calculated slope and intercept values for NO2 in the NOx calibration worksheet have to be entered into the ESC 8832 and the worksheet for the upcoming Zero/Span/Precision check.
- 2.4.5.11 Refer to Section 11, Data logger 8832 SOP.

In the ESC 8832 return to the Main Menu (use the Esc button). Press C (Configuration Menu), D (Configure (Data) Channels), C (Change Old Configuration). Use arrows to skip to NO, press Enter. Use arrow keys to skip to 'Slope=High output (E.U.s)' and enter the 'DAS Regression Data NO DAS Slope X1Y1' from the instrument logbook calibration worksheet (Fig. 8). Use arrow keys to skip to 'Intercept=Low output (E.U.s)' and enter the 'DAS Regression Data NO DAS Intercept X1Y1' from the instrument logbook calibration worksheet (see Appendix: 'NOx Calibration worksheet' for assistance). Use the arrow keys to skip to 'Finished (Config. Now)' and press Enter to save changes.

Repeat this procedure for the NO_x channel using the 'DAS Regression Data NO_x DAS Slope/Intercept X2Y1' values.

Repeat this procedure for the NO₂ channel using the 'DAS Regression Data NO₂ DAS Slope/Intercept XY' values.

These new slopes and intercepts will apply to all future NO-NOx data until the next adjusted calibration. Note time and date new slope and intercept were entered into the datalogger on logbook calibration worksheet.

- 2.4.5.12 In the instrument logbook, create a Z/S/P worksheet (Fig. 18) and enter the new 'DAS Regression Data NO DAS Slope/Intercept X1Y1'; 'DAS Regression Data NO_x DAS Slope/Intercept X2Y1'; 'DAS Regression Data NO₂ DAS Slope/Intercept XY' and 'API Regression Data NO DAS Slope/Intercept X1Y1'; 'API Regression Data NO_x DAS Slope/Intercept X2Y1'; 'API Regression Data NO₂ DAS Slope/Intercept XY' from the calibration worksheet into the Z/S/P worksheet (Fig. 18). This will have a new Z/S/P worksheet ready for the next check due 14 days after the completion of the calibration.
- 2.4.5.13 In the ESC 8832 skip back to the Main Menu (use Esc). Press C (Configuration Menu), D (Configure (Data) Channels), C (Change Old Configuration). Press E (Enable/Mark Channel Online). Use arrows to skip to NO-NO2-NOx, then press Enter for each to enable the all three channels. Refer to Section 11, Data logger 8832 SOP.
- 2.4.5.14 Record a note in the ESC 8832 data logger logbook and the digitrend graph where the adjusted calibration was performed.
- 2.4.5.15 Verify the sample line is connected to the NOx solenoid, which leads to the sample port of the NOx analyzer.
- 2.4.5.16 Close all APICOM windows to disconnect from the NO2 Analyzer.

2.4.6 NO/NO2/NOx 90-Day Verification

The 90-day Verification procedure is similar to the Adjusted Multi-point Calibration procedure (see section 2.4), with the exception of <u>no adjustments are made to any equipment and engineering units are used from the 8832 instead of voltages</u>. The procedure is also divided, as in the Adjusted Multi-point calibration, into Short Path (no ozone introduction) with only the NO-NO_x values recorded, Presets and the Gas Phase Titration (GPT) during which ozone is introduced to the calibration gas to record NO-NO_x-NO₂ values. A 10% difference is also used as the acceptable limit in the verification instead the 2% difference used in the calibration. The NO₂ 90-day worksheet (Fig. 13) is to be used for the verification. The converter efficiency (CE) is also verified to check the moly converter's operation. The CE needs to be .9600 >= CE <= 1.0200. If not, inform the Program Manager.

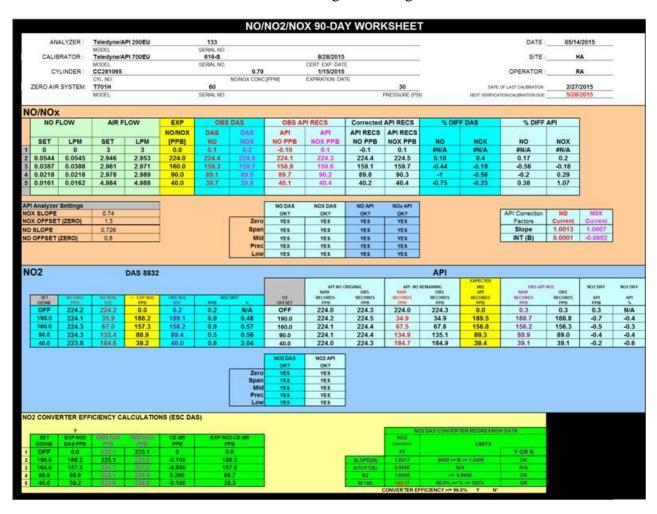


Figure 13: 90-day Verification Data Worksheet

2.5 Teledyne API 700EU Dynamic Dilution Calibrator

In ambient air monitoring applications, precise gas mixture concentrations are required for the calibration of NO2 analyzers. Gas cylinder standards must be certified and used before the certification expires. The Mass Flow Controllers (MFC) must also be certified and if need be, calibrated every 6 months. Pressure transducers must also be verified and if need be, adjusted every 6 months.

A Teledyne API 700EU Dynamic Dilution Calibrator is used to generate ozone to produce calibration gas for NO₂ calibrations in the network. This calibrator is also used to calibrate an Ozone analyzer located at the same site so it must be kept as a true level 3 transfer standard. Normally the NO2 alone does not require the calibrator's photometer to be certified. A Teledyne API T700U Dynamic Dilution Calibrator is used to audit the T-API 200EU NO-NO_x-NO₂ analyzer in the network. Currently our network utilizes two level 2 transfer standards: a Teledyne API 703E Photometric Ozone Analyzer (SN 59) (bench primary standard) and a Teledyne API T700U Dynamic Dilution Calibrator (transfer primary standard). The SN 59 and T700U are verified annually against a standard reference photometer (EPA Region 4 SRP#10) and all previous verifications (up to 6, if available) are used to calculate the SN 59 and T700U verification equation. The SN 59 and T700U are verified by USEPA Region 4 annually, in accordance with USEPA Region 4 procedure.

For verification and maintenance procedures of the Teledyne API 700EU Dynamic Dilution Calibrator, refer to Section 12 Standard Operating Procedure (SOP) Calibrators.

2.6 Teledyne API 701H Zero Air Generator, Teledyne API T701H Zero Air Generator

A zero air system to be used in the field should be constructed as follows: a zero air generator, a valve connected to the output that is connected to two drying columns filled with fresh silica gel followed by a column of activated charcoal containing a layer of Purafil, the air is then passed through a 5 μ m teflon filter to remove particulate.

- 2.6.1 A check of the zero air system should be performed annually.
- 2.6.2 Annually, the entire zero air system, including the zero air generator and drying columns, should be brought back to the laboratory.
- 2.6.3 At this time, replenish the drying column with fresh silica gel, activated charcoal, and fresh Purafil. Replace the filter at this time.
- 2.6.4 Replace the filter on rear of zero air generator. Check the canisters for leaks before re-installing them into the generator.
- 2.6.5 After the annual maintenance is completed, attach the zero air to a flow certified calibrator.
- 2.6.6 Prepare to run a zero point with the calibrator to an analyzer.

2.6.7 Let the analyzer stabilize and observe the ozone value which should read \pm .002 ppm of zero. If not, contact the Program Manager for how to proceed.

For maintenance procedures, refer to Section 13 Standard Operating Procedure (SOP) Zero Air Supplies.

2.7 Teledyne API 200EU Nitrogen Oxide Analyzer Maintenance

On a regular schedule (yearly), the analyzer should be inspected to assure proper functionality. If the instrument is malfunctioning or breaks down, immediate checks and repairs are to be performed. Perform yearly inspections and filter changes AFTER a QC check passes.

Record all maintenance in the logbook.

Before turning the instrument off, check the diagnostics by using the test button on the front panel display. If there are any discrepancies to the manufacturer's specifications (see manual) they should be addressed first.

- 2.7.1 Clean the sample line annually or as necessary. If the sample line becomes extremely dirty replace the line.
- 2.7.2 Replace the 5 µm Teflon particulate filter at least monthly (Fig 14).



Figure 14: Sample box filter housing

A good habit is to replace it after every other bi-weekly Z/S/P check. The filter may be replaced more often if necessary. Refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual (May 2010), chapter 13.3.1.

2.7.3 Replace the Ozone Dryer Particulate filter. Refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual, Chapter 13.3.2.

- 2.7.4 Clean the Reaction Cell. Refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual, Chapter 13.3.9.
- 2.7.5 Inspect and clean the Thermoelectric Cooler Fins. The cooler fins on the PMT Cooler should be inspected and cleaned at six-month intervals. This assures optimal performance of the cooler.
- 2.7.6 Perform a sample vacuum leak and pump check, refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual, Chapter 13.3.12.1. If the leak check fails, perform necessary maintenance. To rebuild the external sample pump, refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual, Chapter 13.3.4.
- 2.7.7 Verify the sample flow is at 1000 ± 50 cm³ and the Ozone flow is at 80 ± 10 cm³, refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual, Chapter 10.7 and 13.3.12.3. If either flow is bad, rebuild the faulty critical flow orifice. Refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual, Chapter 13.3.10.
- 2.7.8 Check the HVPS value on the front display (use the test buttons). The HVPS has to be between 400-900V. If it is outside this range, a PMT Adjustment is necessary, refer to Teledyne API Service Note 13-002 'Performing a PMT Adjustment on a NO/NO_x Analyzer'.
- 2.7.9 Perform a Moly Converter Calculation (see section 2.2.3.6). If necessary replace the Moly Converter, refer to Teledyne API 200E Nitrogen Oxide Analyzer Manual, Chapter 13.3.8.
- 2.7.10 Record all maintenance performed on the instrument in the preventive maintenance logbook located on the S: Drive (Fig. 15).

Preventive Maintenance Log for a 200EU			Maintenance Performed and completed		
Site: Serial #:	900 di		Date	Ву	Notes
Check all electrical connec	tions				1
Check all pneumatic conne	ctions				
Clean PMT cooling fins					
Check vacuum pump and re	epair				
Replace sintered filter and '	'O" rings and cle	an critical orifice			
Clean Reaction Cell					
Turn unit ON and allow it to	warm up (~30m)				
perform leak check	1 1000				
Calibrate flow					
Calibrate pressure					
Calculate Moly Converter E	fficiency				
Adjust Analog Outputs					
Run test points from calibra	tor to test NOx r	eadings			

Figure 15: Preventive maintenance logbook for 200EU

2.8 Routine site visits

The purpose of the routine site visit is to ensure the site is safe, sampling is undisturbed and the 200EU analyzer and ESC 8832 data logger are operating properly. Routine site visits should be conducted at least weekly and more frequently if necessary.

- 2.8.1 Upon arrival, visually inspect the site for safety hazards and cleanliness. Ensure the sample line and probe box are intact, and that there are no problems such as tree branches or other greenery growing too close to the sample inlet, etc.
- 2.8.2 Inspect the building interior for cleanliness of instruments, PC, desk and floor. Check the AC and heater systems for proper function and make sure there are no safety hazards.
- 2.8.3 Check and ensure that the sample line is connected to the back of the analyzer and that it is not contaminated by dirt or moisture. Check electrical connections for proper seating.
- 2.8.4 Check to see that the computer and the ESC 8832 data logger are set to eastern standard time. If times are off more than 1 minute, adjust to correct time, make a note in the logbook and alert program manager.
- 2.8.5 Record the site visit in the Agilaire EDAS logbook (Fig 16a and b).

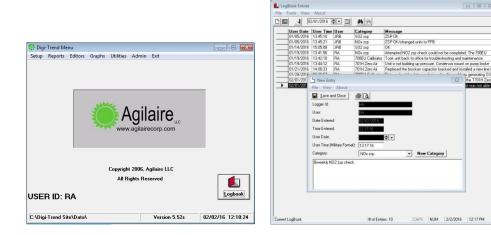


Figure 16a/16b: Agilaire EDAS logbook entry

- 2.8.6 Examine the entire ESC 8832 data logger graph day by day since the last visit and check for atypical data. Record any discrepancies on the graph and in the logbook if necessary.
- 2.8.7 Check the frequencies, pressure, and temperature (see 2.2.3.4) on the instrument. Ensure that the hourly data is typical and that the previous day's auto-calibration cycle is ok. Corrective action should be taken if the zero is $> \pm 5$ ppb NO-NO_x-NO₂ or the span or precision is $> \pm 8$ ppb NO-NO_x-NO₂ from the expected value. Corrective action must be taken if the zero is $> \pm 8$ ppb NO-NO_x-NO₂ or the span or precision is $> \pm 10$ ppb NO-NO_x-NO₂ from the expected value. Corrective actions that may be performed are:

- 1. Inform Program Manager
- 2. Repair/Maintenance
- 3. Repeat the auto-calibration
- 4. Perform a Bi-weekly Zero/Span/Precision (ZSP) check (2.9.5).
- 5. Perform an Adjusted Multi-point Calibration

2.8.8 Any possible abnormalities should be investigated to ensure continuous uninterrupted quality controlled data collection. If any problems are found the operator is to notify the program manager and do whatever is necessary to permanently correct the problem. If the operator is not absolutely sure the problem encountered is permanently rectified, he should revisit the site later on that day or the next working day to check the problem. The operator is to keep the supervisor informed on a daily basis as to the status of the problem. Detailed records of all corrective actions are to be maintained in the ESC 8832 datalogger, graph and site logbooks (EDAS & Excel).

2.9 Quality Assurance/Quality Control checks

Quality Assurance (QA)/Quality Control (QC) procedures include performance audits, 90-day verification checks, zero-span-precision (ZSP) 14-day checks, and calibration checks.

Analyzer accuracy audits are to be performed once for each quarter of the year and by an individual other than the operator who performed the calibration. The QA staff performing the audit should also inspect the site's overall condition and report any issues to the Program Manager. Issues can include but not be limited to: safety hazards, operator oversights, EPA site requirements being met, building condition, overall neatness, and up-to-date documentation of the site's activities. The audit should be conducted using a gas dilution system, certified NIST EPA protocol cylinder gas and zero air system that are **independent of the normal calibration system**.

2.9.1 Audit Short Path Procedure

Audits are to be performed quarterly at a frequency <= 90 days apart. Analyzer accuracy audits are to be performed by an individual other than the analyst who performed the calibration. The audit should be conducted using a gas dilution system, certified NIST EPA protocol cylinder gas, and zero air system that are independent of the normal calibration system. The following procedure should be followed when conducting audits.

2.9.1.1. The analyzer audit is conducted by challenging the measurement system with a series of known concentrations of calibration gas. The audit field procedure is similar to the 90-day verification procedure except that different points can be run. The NO-NOx points of the audit must be the following 5 points: a zero, 225 ppb, 160 ppb, 70 ppb, and 40 ppb.

The NO2 portion of the audit must include zero and at least one point taken from three of the ten ranges:

Level 1: 0.3-2.9 ppb NO₂ (Required)

Level 2: 3.0-4.9 ppb NO₂ Level 3: 5.0-7.9 ppb NO₂ Level 4: 8.0-19.9 ppb NO₂

Level 5: 20.0-49.9 ppb NO₂ (Required)

Level 6: 50.0-99.9 ppb NO₂

Level 7: 100.0-299.9 ppb NO₂ (Required)

Level 8: 300.0-499.9 ppb NO₂ (Over FCEAP range)

Level 9: 500.0-799.9 ppb NO₂ (Over FCEAP range)

Level 10: 800.0-1000.0 ppb NO₂ (Over FCEAP range)

2.9.1.2 The audit is to be recorded in the audit section of the analyzer logbook. Figure 17 should be used for a template of data that must be recorded in the logbook. Record the information but do not alter the analyzer settings in any way.

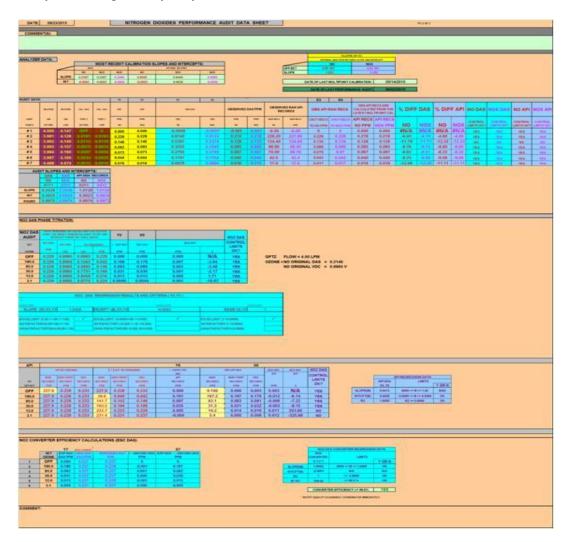


Figure 17: Audit Data Worksheet

2.9.1.3 Transport an audit dynamic calibration system (i.e. Teledyne API T700U Dynamic Dilution Calibrator), an audit gas certified by EPA traceable to NIST standards, and an independent zero air

Section 2 SOP for NO₂ May 2016 Page 34 of 47 Revision 1

system to the site to be audited. The audit calibrator may be transported to the site the day before the audit if feasible. The audit calibrator should warm up at least one hour prior to the performance of the audit. The audit calibrator's mass flow controllers must be calibrated against authoritative standards such as an NIST traceable bubble meter, a wet test meter or a calibrated BIOS Drycal prior to use and should be recertified semi-annually. The calibration slope and intercept prepared when calibrating the audit calibrator will be used to determine calibrator flows.

- 2.9.1.4 Use a pump (oil-less diaphragm or oil-less piston type) to supply a source of audit zero air. The pump should be capable of supplying at least 20 psig at 10 lpm. The audit zero air should be dried with silica gel or drierite, passed through canisters containing purafil and charcoal and filtered through a 5 µm particulate filter prior to entering the audit calibrator.
- 2.9.1.5 Connect 1/4" O.D. FEP Teflon tubing from the audit zero air system to the audit calibrator zero air in port.
- 2.9.1.6 To Login into the ESC 8832, open 'HyperTerminal' on the PC.

Refer to Section 11, Data logger 8832 SOP.

Press L (Login), type password, press Enter. C (Configuration Menu), D (Configure (Data) Channels), C (Change Old Configuration). Press M (Disable/Mark Channel Offline). Use arrows to skip to NO-NO2-NOx, then press Enter for each to disable the all three channels.

- 2.9.1.7 Connect the audit calibrator output with 1/4" Teflon tubing to the analyzer sample line inlet. The length of the tubing should be kept to a minimum. An atmospheric vent should be utilized. The normal sample setup is through an ambient solenoid and particulate filter so the audit should be setup similarly.
- 2.9.1.8 Attach a two-stage regulator to the audit NO cylinder. Quickly open and close the cylinder valve on the NO audit cylinder and adjust the 1st stage regulator valve to 24 psig. Open the second stage valve and allow the regulator to empty. Close the second stage valve. Repeat this process 5 times to evacuate residual gases in the regulator. **The regulator evacuation should be performed in a well ventilated area.** After the evacuation procedure fill the regulator with gas leaving the second stage valve closed. Connect the NO regulator to the audit calibrator with the appropriate tubing (stainless steel) and fitting (stainless steel). Open the second stage regulator valve to the maximum. Adjust the second stage pressure to 24 psig. Check the fittings for leaks with Snoop[®]. Record the cylinder pressure.
- 2.9.1.9 Switch on the audit zero air pump. Set the audit calibrator for an output of 0.000 ppm NO-NO_x-NO₂ by pressing SEQ and toggle to ZERO and press enter.
- *During the Short Path part of the audit, no ozone is introduced and only the NO-NO_x values are recorded. The only NO₂ value used is the zero point NO₂ observed 8832 and 200EU reading.
- 2.9.1.10 Check the instrument functions by pressing the Test button. Check the analyzer temperatures, pressure, flow, and intensities.
- 2.9.1.11 Allow the instrument to stabilize (~30 minutes). Observe the analyzer and the calibration standard. The difference for the zero point should meet the following specification:

Section 2 SOP for NO₂ May 2016 Page 35 of 47 Revision 1

 $\leq \pm 5$ ppb at a stability of < 0.5

- 2.9.1.12 Record (highlight, right click 'copy') the ppb reading from the ESC 8832 (in Hyperterminal) and download the instrument's 1-minute readings using the APICom software. Paste both, the data logger (ESC) and copy/paste APICom min data, readings into the audit data worksheet (Fig. 17) in the correct cells.
- 2.9.1.13 While the zero point is still running, on the calibrator check the NO Flow and Air Flow settings and actual flows. Type these readings to the NO FLOW Set/Lpm and AIR FLOW Set/Lpm in the worksheet. Check the expected NO/NO_x values and type it into the EXP NO/NOx [PPB] in the worksheet.
- 2.9.1.14 Press SEQ on the calibrator, use the arrow keys to reach NO_x225 , press Enter to start the Span point.
- 2.9.1.15 Allow the instrument to stabilize (15-30 minutes). Observe the analyzer and the calibration standard. The difference for the span point should meet the following specification:
 - $\leq \pm 10\%$ difference at a stability of < 0.5
- 2.9.1.16 Record the raw voltage reading from the ESC 8832 and download the instrument's 1-minute reading using APICom. Paste (ESC) and copy/paste (APICom) both readings into the audit data worksheet (Fig. 17).
- 2.9.1.17 While the span point is still running on the calibrator, check the NO FLOW Set/Lpm and AIR FLOW Set/Lpm settings and the EXP NO/NO_x [PPB] values and type them into the correct cells in the audit data worksheet (Fig. 17).
- 2.9.1.18 After the 0.0 ppb (Zero) and 225 ppb (Span) NO-NO_x points have been run satisfactorily and data recorded, start the sequences on the calibrator to run the points for NO_x 160 ppb, NO_x 90 ppb, and NO_x 40 ppb. Press SEQ on the calibrator, use arrow keys to reach the desired NO-NO_x concentration, press Enter.

Record the results for each concentration based on stable readings (stability <0.5) from the ESC 8832 ppb readings and 1-minute APICom (see 2.4.2.5) in the audit data worksheet (Fig. 17) and manually copy them into the correct cells. The difference for each point should meet the following specification:

 $\leq \pm 10\%$ difference

If it is not within the % difference for each point inform the Program Manager.

Document the NO FLOW Set/Lpm and AIR FLOW Set/Lpm setting and the EXP NO/NO_x [PPB] values in the audit data worksheet (Fig. 17).

Section 2 SOP for NO₂ May 2016 Page 36 of 47 Revision 1

2.9.2 Ozone Presets for Audits (GPTPS)

2.9.2.1 Before continuing the audit procedure for NO2 with the Gas Phase Titration (GPT) part, presets have to be run on the audit calibrator. Press SEQ on the calibrator, use arrow keys to reach EXE.SEQ.PS, press Enter.

The preset mimics the calibrator set up for running the following GPT without mixing any O3 with calibration gas. Instead, the internal photometer measures the actual ozone concentration and adjusts the ozone drive voltage on the ozone generator, to receive a most accurate NO₂ reading later during the GPT.

The preset will run approximately 15-20 min and no records have to be taken during the preset, as this only prepares the calibrator for the following GPT. Observe the 'Active' and 'Auto' lights on the front panel. While both lights are blinking the calibrator is adjusting the ozone drive voltage. When the 'Active' light is steady lit, it's setting a reference point. There will be a total of 8 steps to complete the presets sequence.

Once the calibrator is back in Standby mode, proceed with the Gas Phase Titration Zero (GPTZ) procedure.

2.9.3 NO-NOx GPTZ for Audits Explanation

During the GPTZ, ozone is not introduced to the calibration gas mixture but the flow paths and amounts follow the GPT settings for a given desired result. The GPTZ steps will produce the NO_{orig} and NOx_{orig} (original) values used along with NO_{rem} and NOx_{rem} (remaining) collected during the GPT steps to calculate expected NO2 levels.

- 2.9.3.1On the calibrator press SEQ, use the arrows to reach GPTZ, press Enter. This will start the zero point for the GPT run. This GPTZ point will mimic the flow settings for the 190 ppb NO2 GPT point but will serve as the zero point for NO2. The results can be used as the NO_{orig} and NOx_{orig}.
- 2.9.3.2 Allow the instrument to stabilize (15-30 minutes). Observe the analyzer and the calibration standard. The % error for the GPTZ points should meet the following specification:
 - $\leq \pm 10\%$ difference of 225ppb at stability of < 0.5
- *Note: Each GPT point that will be run will have a GPTZ point ran right before it with the same flow and desired ozone level targets as the GPT.
- 2.9.3.3 Copy the ppb readings from the ESC 8832 and download the instrument's 1-minute reading using APICom (see 2.4.2.5). Copy both readings into the audit data worksheet (Fig. 17). The following observed DAS and API NO, NO_x and NO₂ readings have to be manually copied into the correct cells.

2.9.4 NO2 GPT for Audits

- 2.9.4.1 On the calibrator press SEQ, use the arrows to reach NO2 190 (Span point), press Enter.
- 2.9.4.2 Allow the instrument to stabilize (15-30 minutes). Observe the analyzer and the calibration standard. The difference for all NO2 points should meet the following specification:
 - $\leq \pm 10\%$ difference of the calculated expected ppb for NO2 at stability of < 0.5
- 2.9.4.3 Copy the ppb reading from the ESC 8832 and download the instrument's 1-minute reading using APICom (see 2.4.2.5). Copy both readings into the audit data worksheet (Fig. 17). The following observed DAS and API NO, NO_x and NO₂ readings have to be manually copied into the correct cells of the audit data worksheet (Fig. 17).
- 2.9.4.4 After the 0.00 ppb (Zero) and 190 ppb (Span) GPT points have been run satisfactorily and recorded, start the sequences on the calibrator to run points for GPT NO₂ 160, GPT NO₂ 70 ppb and GPT NO₂ 40 ppb.
- *Before each of the following GPT points are to be run, a GPTZ point has to be run with the same target ozone and total flow to be used for each of the GPT points. During the GPTZ 160, GPTZ 70 and GPTZ 40 the NO_{Orig} and NO_{xOrig} are obtained to get more accurate NO_{Rem} and NO_{2Rem} calculations. On the calibrator press SEQ, use the arrow keys to reach the desired GPTZ point. See section 2.9.3.
- 2.9.4.5 Record the results for each concentration based on stable readings on the analyzer (stability <0.5) and using the digitrend chart. Copy the ppb readings from the ESC 8832 and the instrument's 1-minute reading using APICom (see 2.4.2.5) in the audit data worksheet (Fig. 17) and manually copy into the corresponding into the correct cells. From each associated GPTZ run, manually copy the API NO_{Orig} and DAS NO_{xOrig} values into the correct cells.
- 2.9.4.6 After all NO2 points are done verify that each result has a difference \ll 10%. If not, inform the Program Manager. The converter efficiency (CE) is also verified to check the moly converter's operation. The CE needs to be $.9600 >= CE \ll 1.0200$. If not, inform the Program Manager.
- 2.9.4.7 Close all APICom windows to disconnect from the NO2 Analyzer.
- 2.9.4.8 If the sample line was disconnected, reconnect the sample line to the sample port of the analyzer.
- 2.9.4.9 On the calibrator press the STBY button to bring it back in standby mode. Check the analyzer for it to return to reading ambient NO-NO_x-NO₂ values.
- 2.9.4.10 In the ESC 8832 skip back to the Main Menu (use Esc). Press C (Configuration Menu), D (Configure (Data) Channels), C (Change Old Configuration). Press E (Enable/Mark Channel Online). Use arrows to skip to NO-NO2-NOx, then press Enter for each to enable the all three channels.

Refer to Section 11, Data logger 8832 SOP.

2.9.4.11 Record a note in the ESC 8832 data logger logbook and the digitrend graph where the audit was

Section 2 SOP for NO₂ May 2016 Page 38 of 47 Revision 1

performed.

2.9.5 Bi-weekly Zero/Span/Precision Checks (Z/S/P)

Zero/Span/Precision checks (Z/S/P) must be performed every 14 days. The Z/S/P procedure is divided into two parts: Short Path, during which no ozone is introduced and only the NO-NO_x values are recorded, and Gas Phase Titration (GPT) during which ozone is introduced to the calibration gas to record NO-NO_x-NO₂ values. Concentrations for the Short Path points are 0.0 ppb NO_x (Zero), 225 ppb NO_x (Span) and 70 ppb NO_x (Precision). Concentrations for the GPT points are 0.0 ppb NO₂ (Zero), 190 ppb NO₂ (Span) and 70 ppb NO₂ (Precision) respectively. The Z/S/P check must be performed with a currently certified gas dilution system (Teledyne API 700EU Dynamic Dilution Calibrator) and a NIST cylinder gas, which is currently certified according to EPA protocol.

- 2.9.5.1 Make sure the 700EU Calibrator is connected to a source of zero air producing 25-30 psig pressure. Check the regulator pressure on the 700EU Calibrator to make sure it is set to 7-10 psig.
- 2.9.5.2 Check that the 700EU Calibrator is connected to the NO-NO_x-NO₂ analyzer. Make sure the calibration gas passes through all filters, conditioners, and other components used during normal ambient sampling and as much of the ambient air inlet system as is practicable.
- 2.9.5.3 To Login into the ESC 8832, open 'HyperTerminal' on the PC

Refer to Section 11 Data logger 8832 SOP.

Type Esc 'site ID'(HA) AQM Enter, to enter Login screen. Press L (Login), type password, press Enter. D (Real-time Display Menu), O (Display all Digital Outputs). Use arrows to skip to NO-NO_x-NO₂ Bad Stat, press Enter to disable the NO-NO_x-NO₂ channel.

2.9.5.4 Using the APICom software download one instrument's 1-minute reading (see 2.4.9.6) and copy it into the instrument's logbook (200EU Diag., top right) (Fig. 18).

Section 2 SOP for NO₂ May 2016 Page 39 of 47 Revision 1

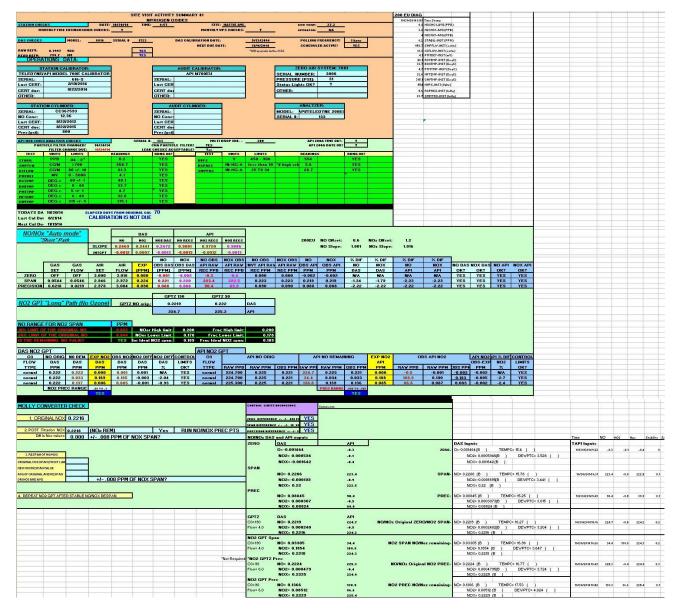


Figure 18: Instrument Logbook, Zero/Span/Precision worksheet

2.9.5.5 In the ESC 8832 skip back to the Main Menu (use Esc). Press D (Real-Time Display), F (Display Readings w/Flags).

2.9.5.6 Short Path Procedure

- 2.9.5.6.1 Start the zero point by pressing SEQ on the 700EU Calibrator, toggle to NOxZERO and press Enter. Let the point run for at least 10 minutes until stability reaches < 0.5.
- 2.9.5.6.2 Use Esc to skip back to the Main Menu. Press D (Real-Time Display), F (Display Readings w/Flags). Copy (highlight, right click 'copy') the ppb readings from the ESC 8832. (Fig 19)

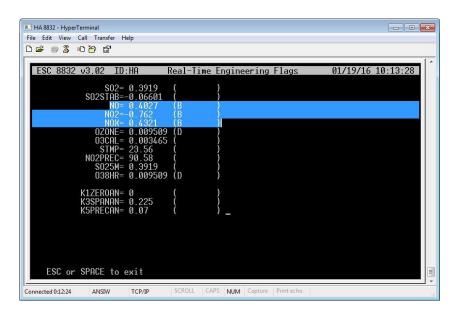


Figure 19: ESC 8832 NO/NO2/NOx ppb Readings

- 2.9.5.6.3Download the instrument's 1-minute readings using APICom (see 2.4.2.5). Paste both readings into the appropriate sections of DAS input (Raw Readings) and TAPI inputs (1-minute reading) in the instruments logbook (Fig. 18). The observed and downloaded NO-NO_x, not NO₂, readings have to be manually copied into 'NO/NOx "Auto Mode" Short Path' section NO_{Obs}/NO_{xObs} DAS and NO_{Obs}/NO_{xObs} API Raw Rec. The NO₂ reading has to be entered into OBS NO2 DAS in the DAS NO2 GPT section (see Appendix C 'NOx Zero/Span/Precision worksheet, data input and handling' for assistance).
- 2.9.5.6.4 While the zero point is still running, on the 700EU Calibrator check the NO Flow and Air Flow settings and actual flows. Type these readings to the NO FLOW Set/Lpm and AIR FLOW Set/Lpm in the worksheet. Also check the expected NO/NO_x values and type it to the EXP NO/NO_x [PPB] in the worksheet.
- 2.9.5.6.5 Start the span point by pressing SEQ, then NOx225 on the 700EU Calibrator. Let the point run for at least 10 minutes until stability reaches < 0.5.
- 2.9.5.6.6 Copy the Reading from the ESC 8832. Download the instrument's 1-minute reading using APICom (see 2.4.2.5). Paste both readings into the appropriate sections of DAS input (Raw Readings) and TAPI inputs (1-minute reading) in the instrument's logbook (Fig. 18). The following observed NO-NO_x, not NO₂, readings have to be manually copied into 'NO/NOx "Auto Mode" Short Path' section NO_{obs}/NO_{xObs} DAS and NO_{obs}/NO_{xObs} API Raw Rec. The NO₂ reading does not have to be manually copied (see Appendix 'NOx Zero/Span/Precision worksheet, data input and handling' for assistance).

From the 700EU Calibrator, type the NO FLOW Set/Lpm, AIR FLOW Set/Lpm, and EXP NO/NOx [PPB] readings.

2.9.5.6.7 Start the precision point by pressing SEQ, then NOx90 on the 700EU Calibrator. Let the point run for at least 10 minutes until stability reaches < 0.5.

Section 2 SOP for NO₂ May 2016 Page 41 of 47 Revision 1

2.9.5.6.8 Copy the reading from the ESC 8832. Download the instrument's 1-minute reading using APICom (see 2.4.2.5). Paste both readings into the appropriate sections of DAS input (Raw Readings) and TAPI inputs (1-minute reading) in the instrument's logbook (Fig. 18). The observed and downloaded NO-NO_x, not NO₂, readings have to be manually copied into 'NO/NOx "Auto Mode" Short Path' section NO_{Obs}/NO_{xObs} DAS and NO_{Obs}/NO_{xObs} API Raw Rec. The NO₂ reading does not have to be manually copied (see Appendix 'NOx Zero/Span/Precision worksheet, data input and handling' for assistance).

From the 700EU Calibrator, type the NO FLOW Set/Lpm, AIR FLOW Set/Lpm, and EXP NO/NOx [PPB] readings.

2.9.5.6.9 The difference for each point should meet the following specification:

 $\leq \pm 10\%$ difference

If it is not within the % difference for each point inform the Program Manager.

2.9.5.7 Before continuing with the (GPT) procedure, a preset run can be executed on the 700EU Calibrator. It is only required if the NO₂ values from previous Z/S/P checks show an increase in error, i.e. drift. The preset mimics the 700EU Calibrator set up for running the following GPT points without introducing NO to the calibration gas. Instead, the internal photometer measures the actual ozone concentration and adjusts the ozone drive voltage on the ozone generator, to receive a most accurate NO₂ reading during the GPT afterwards. Typically the presets should be run about every two months.

To start the preset, press SEQ on the 700EU Calibrator, use arrow keys to reach EXE.SEP.PSZSP, press Enter.

The preset will run approximately 12-15 min and no records have to be taken.

Once the 700EU Calibrator is back in Standby mode, proceed with the GPT procedure.

2.9.5.7.1 GPTZ Procedure

- 2.9.5.7.2 Start the GPT zero point by pressing SEQ, then GPTZ on the 700EU Calibrator. Let the point run for at least 10 minutes until stability reaches < 0.5.
- 2.9.5.7.3 Copy (highlight, right click 'copy') the ppb reading from the ESC 8832. Download the instrument's 1-minute reading using APICom (see 2.4.2.5). Paste both readings into the appropriate sections of DAS input (ppb) and TAPI inputs (1-minute reading) in the instrument's logbook (Fig. 18). The following observed NO, not NO_x or NO₂, readings have to be manually entered into NO Rem DAS in the DAS NO2 GPT section and API NO Rem. The NO₂ readings have to be manually copied into Obs API NO2 in the DAS NO2 GPT section (see Appendix 'NOx Zero/Span/Precision worksheet, data input and handling' for assistance).

Also manually copy the DAS and API NO value into "NO2 GPT "Long Path" (No Ozone) GPTZ190.

2.9.5.7.4 Check the Moly Converter Check Original NOx.

- 2.9.5.7.5 Start the GPT span point by pressing SEQ, then NO2 190 on the 700EU Calibrator. Let the point run for at least 10 minutes until stability reaches < 0.5.
- 2.9.5.7.6 Copy the Reading from the ESC 8832. Download the instrument's 1-minute reading using APICom (see 2.4.2.5). Paste both readings into the appropriate sections of DAS input (ppb) and TAPI inputs (1-minute reading) in the instrument's logbook (Fig. 18). The following observed NO and NO₂, not NO_x, readings have to be manually copied into NO Rem DAS and Obs NO2 DAS and API NO Rem Raw and Obs API NO2 in the DAS NO2 GPT section (see Appendix C 'NOx Zero/Span/Precision worksheet, data input and handling' for assistance).
- 2.9.5.7.7 Start the GPT precision point by pressing SEQ, then NO2_90 on the 700EU Calibrator. Let the point run for at least 10 minutes until stability reaches < 0.5.
- 2.9.5.7.8. Copy the Reading from the ESC 8832. Download the instrument's 1-minute reading using APICom (see 2.4.2.5). Paste both readings into the appropriate sections of DAS input (ppb) and TAPI inputs (1-minute reading) in the instruments logbook (Fig. 18). The following observed NO and NO₂, not NO_x, readings have to be manually copied into NO Rem DAS and Obs NO2 DAS and API NO Rem Raw and Obs API NO2 Raw in the DAS NO2 GPT section (see Appendix C 'NOx Zero/Span/Precision worksheet, data input and handling' for assistance).
- 2.9.5.8 Check that Zero/Span/Precision Differences are within a 10% difference.

If the biweekly check does not meet the above criteria, check the instrument line set up. If there are no issues in the set up, inform the Program Manager.

- 2.9.5.9 When finished, press the STBY button on the 700EU Calibrator to set into Stand By mode. Make sure the front of the analyzer is showing it is in 'Sample' mode; observe the NO-NO_x-NO₂ values and stability to make sure it is returning to ambient values.
- 2.9.5.10 Once a month the 5 µm filter has to be changed. This has to be done <u>after</u> the Biweekly Zero/Span/Precision Check was performed. Always handle particulate filters with tweezers.
- 2.9.5.11 After every Biweekly Zero/Span/Precision Check a record of all hourly data of the past 14 days has to be downloaded. In APICom 4.0.1exe. click the 'Get data' button, choose 'since last download (15 days)' to download the hourly data from the 200EU and 'Save' to the NOx hourly data folder (see 2.4.9.6). Close all APICom windows to disconnect from 200EU.
- 2.9.5.12 Go to the ESC 8832 data logger and enable the NO-NO_x-NO₂ channel.

Refer to Section 11 Data logger 8832 SOP

Using 'Ecs' skip back to Main Menu. Press D Real-time Display Menu, O Display all Digital Outputs. Use arrows to skip to NO-NO_x-NO₂ Bad Stat, press Enter to enable the NO-NO_x-NO₂ channel. Skip back to the Main Menu (Esc), press O Log Out/Exit to exit out of the ESC 8832.

2.9.5.13 Record a note in the ESC 8832 data logger logbook and graph of the performed check.

Section 2 SOP for NO₂ May 2016 Page 43 of 47 Revision 1

2.9.6 Teledyne 200EU Analyzer Nightly Auto-Calibrations

Each night an auto-calibration is triggered by the datalogger and controlled by the calibrator to further test and confirm the equipment's operational status. This program starts at a selected time when ambient readings for a specific pollutant are at their lowest point in a typical diurnal pattern. The datalogger triggers the auto-cal to start but then hands over control to the calibrator. The timing of the check for both the datalogger and calibrator are in sync so the datalogger can capture expected results from the calibrator and the analyzer at the right moment. These records are marked as calibrations and the results are reported to AQS as QC checks. All auto-cals run at least a zero point and a precision point (a point near the current standard) but could include additional points if needed. Auto cal results have to meet the 10% difference. If it does not meet this then corrective action is required. Some troubleshooting may be needed if results are greater than 7% so data loss can be avoided. Most auto-cals are also programmed so reportable hours are not lost in the process. Operators and staff review the results of the auto-cals every workday since all values show up on a daily report (see Data Handling and Reporting SOP section 10).

2.10 Data Handling - Documentation, Reduction, Analysis, and Reporting.

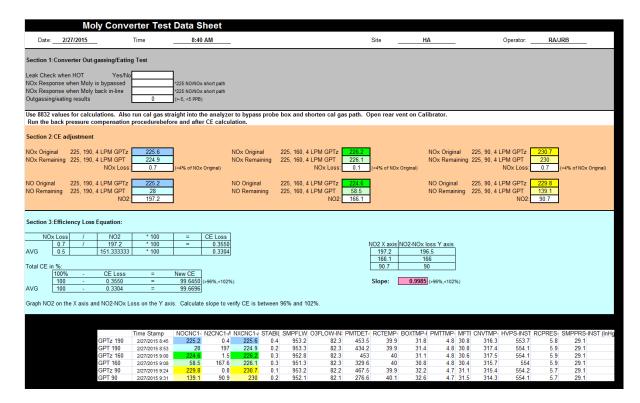
See Section 10, Data Handling and Reporting SOP

Section 2 SOP for NO₂ May 2016 Page 44 of 47 Revision 1

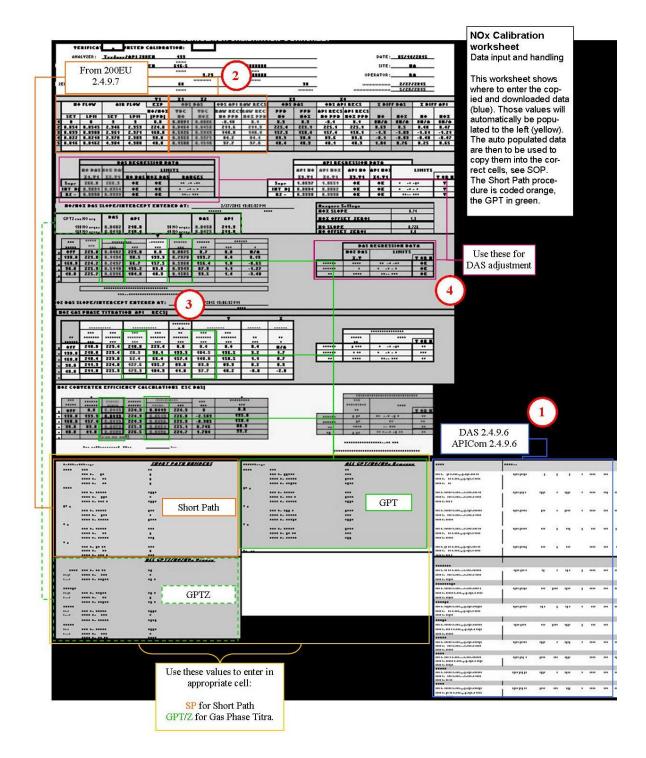
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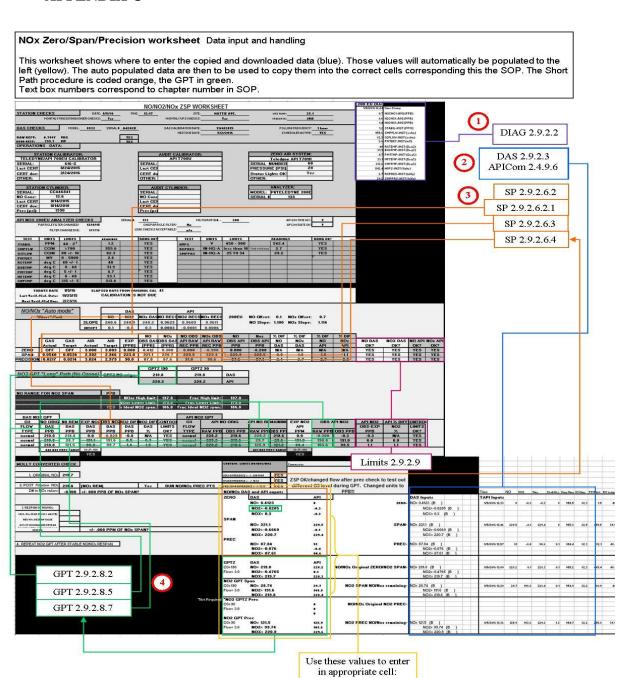
APPENDIX A



APPENDIX B



APPENDIX C



SP for Short Path GPT for Gas Phase Titra.